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INTERLABORATORY STUDY 92-1

POLYCHLORINATED BIPHENYL (PCB) ISOMERS STANDARD SOLUTIONS

IN SUPPORT OF

THE INTEGRATED
ATMOSPHERIC
DEPOSITION
NETWORK
(IADN)

SEPTEMBER 1992

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1 SUMMARY OF INTERLABORATORY STUDY 92-1

Interlaboratory Study 92-1 was initiated in support of the Integrated Atmospheric Deposition Network (IADN) to provide an initial assessment of between-laboratory variability for the analysis of Polychlorinated Biphenyl (PCB) isomers in precipitation and/or ambient air. Participation was limited to laboratories which contribute to the IADN database or related programs. This study was sponsored by the Canada-Ontario Agreement (COA) Air Toxics Workgroup, and conducted as a joint project between the Atmospheric Environment Service (AES) of Environment Canada and the Quality Management Unit (QMU), Laboratory Services Branch (LSB) of the Ontario Ministry of Environment and Energy (MOEE).

Six participating laboratories received a set of four ampouled standards that were ready for direct instrumental analysis. The parameter list consisted of 75 different PCB isomers. Ampoules 1, 2, and 3 contained subsets of the total target list and ampoule 4 contained all 75 isomers. One participant only reported a total value for each congener group. The remaining participants reported results for the individual isomers, though none of the participants had the capability of reporting results for every isomer on the target list.

The results from this study demonstrated interlaboratory means and medians that appear to agree with the target for PCB isomers that have three or more chlorine atoms. However, between-laboratory variability is frequently at a range of 20% or greater, which indicates poor agreement among the participants for may of the PCB isomers. This may introduce greater biases to the IADN database than may be acceptable. The use of a common reference standard by the participants should reduce this source of variability.

The participants demonstrated problems with the analysis of the mono- and dichlorinated biphenyls. As these are more volatile compounds, sample losses may have occurred at the GC injection port. Differences in standards may also have contributed to between-laboratory variability.

Co-elution of PCB isomers also contributed to the between-laboratory variability and affected the accuracy of the results. As technology improves, resolution of different isomer pairs should help reduce between-laboratory variability.

At the time of this study, a final target list of PCB isomers had not been determined for the IADN program. Future interlaboratory studies will focus on the target list of the IADN program and attempt to determine performance criteria. Future studies will not only look at instrumental performance as in this study, but also attempt to address between-laboratory performance on the whole analytical method via sample extracts and spiked matrices.

2 INTRODUCTION

Interlaboratory performance studies are conducted to assess the comparability and accuracy of data among different laboratories. These studies are useful for the identification of biases, precision and accuracy problems. Participation in such studies can serve as a guide for improving individual laboratory performance and maintaining performance standards.

This study was designed to assess the analytical variability among laboratories contributing to the Integrated Atmospheric Deposition Network (IADN). IADN was established as a joint venture between Canada and the United States under the direction of the International Joint Commission[1]. The intent of IADN is to identify toxic airborne substances in the Great Lakes Basin, and by means of the network, quantify the total and net atmospheric loadings of these contaminants, and define spatial and temporal trends in the atmospheric deposition of these substances. Data from several participating agencies is to be merged into a central database. Comparability of these contributing data sets is an important component of the IADN Quality Assurance Implementation Plan[2]. This interlaboratory study provides information on the laboratory component of between agency differences, and can be used to help establish the comparability of the data sets. It is a recommended activity of the IADN Quality Assurance Program Plan[3]. Sponsorship of this interlaboratory study was through the Canada-Ontario Agreement (COA) Air Toxics Workgroup. Funding for the purchase of materials came from the Atmospheric Environment Service (AES) of Environment Canada. Co-ordination and implementation of the study was done by the Quality Management Unit (QMU) of Laboratory Services Branch (LSB) of the Ontario Ministry of Environment and Energy (MOEE).

Interlaboratory Study 92-1 targets laboratories analyzing for Polychlorinated Biphenyl (PCB) isomers in precipitation and/or ambient air. A target list of 75 PCB isomers was chosen for this study, comprising target lists from several contributing agencies. The aim of this study was to establish the comparability of instrumental calibration among the participating laboratories. Each participant received a set of ampouled standards ready for direct instrumental analysis. Ampoules 1, 2, and 3 contained subsets from the target list, while Ampoule 4 contained all of the parameters in the target list.

A list of participants is given in Appendix 2. Each participant was assigned a unique identification code for ease in data manipulation.

Section 3 describes sample preparation, sample distribution, analytical methodology, and data evaluation procedures. Final results are tabled in Appendix 1 and discussed in Section 4.

3 PROCEDURE

3.1 Preparation of Ampouled Standards

Neat PCB isomers of 99%+ purity were purchased from Ultra Scientific and AccuStandard by AES. All subsequent work was done by the QMU of LSB, MOEE. Concentrated stock solutions of each isomer were prepared in toluene and sealed into 5 mL amber ampoules. The stock concentrations were between 10 to 12 mg/L and verified using gas chromatography/mass spectrometry analysis by an analytical unit at LSB not involved in analysis of ambient air or precipitation. Ampouled solutions were stored in a freezer at -20°C.

Solutions for the interlaboratory study were prepared from the concentrated stock solutions by diluting appropriate aliquots into a combined solution. All combined solutions for the study were prepared in iso-octane. Solutions 1, 2, and 3 were designed to consist of subsets of the complete target list. The isomers were distributed so that there would be approximately an equal number of each congener group in each solution and co-eluters or close eluters (based on relative retention time criteria defined by the National Research Council[4]) were in different solutions. Solution 4 contained all PCB isomers on the target list. Concentration levels were designed to fall in the routine analytical range of most participants. As each isomer was present in two different solutions, one solution had a "low" concentration level and the other a "high" concentration level. All solutions were sealed into 5 mL amber ampoules and stored in a freezer at -20°C until shipped to the participants. The ampoules were labelled IADN PCBs 1-4. In the following Discussion, the ampoules are referred to as Ampoules 1-4.

3.2 Sample Distribution

Samples were packed into styrofoam shipping containers and shipped by Purolator Courier to the participating laboratories. A list of the laboratories receiving sample sets is given in Appendix 2. Samples were shipped on September 28, 1992. A copy of all correspondence is also included in Appendix 2.

3.3 Analytical Methodology

Participating laboratories were requested to analyze the samples using their routine in-house methods used to analyze precipitation and/or ambient air samples for the IADN program. Participants were requested on the report form provided (Appendix 2) to summarize their Instrument and Detector used for the analysis. Information regarding the gas chromatograph column was requested at a later date. All participants were assigned a unique identification code that does not correspond with the order the participants are listed in Appendix 2.

3.4 Data Reporting

Results were submitted to the QMU, LSB in written form. All data were manually entered by laboratory code into an electronic spreadsheet.

The participating laboratories were mailed a copy of the tables of results on February 25, 1993. Two participants reported two data sets, using two different instruments. One participant submitted a revised data set after the release of the tables of results, with an explanatory note provided below in the Discussion. Both values are included in the tables, though the corrected results are used in the evaluation.

The interlaboratory mean, median, standard deviation (SD), and relative standard deviation (%RSD) were calculated for each isomer in each ampoule for which there were 2 or more laboratories reporting results and included in Tables 1-4, Appendix 1. As the data set is small, these calculated values are provided as an approximate indicator of the spread of the data and may not necessarily be statistically correct.

4 DISCUSSION

OVERVIEW OF INTERLABORATORY PERFORMANCE

Laboratory 9211 reported only total congener groups. In the following discussion on the study results, this laboratory is not included. Their performance is evaluated in the section on individual participants' results.

The list of PCB Isomers selected for this study included target isomers in the IADN Implementation Plan[2], isomers on the current target lists of the participating laboratories (based on response forms from the invitation to participate), and a summation of potential toxic isomers or those found in the environment that have been cited in the literature (P. Yang, Internal Memorandum, 1992, LSB, MOEE). A total list of 75 isomers was assembled. Out of this list, 22 isomers were on the target list of all participants (Table 5). There were 3 isomers included in the study for which no results were reported (i.e. they were not on the target list of any of the participants - see Table 5). An additional 3 isomers were on the target list of only one participant (Table 5). Other combinations of PCB Isomers on the target lists of 3, 4, and 5 participants are not included in Table 5.

The interlaboratory mean and median appear to demonstrate good agreement with the target values for most isomers of the tri-chlorinated and higher chlorinated congener groups (PCB16 and up). However, due to the small size of the data set, and the "not normal" distribution of most of the results, the statistical calculations are not reliable indicators of the data quality. There are several cases where the interlaboratory mean is biased particularly low relative to the target due to the "ND" or 0 reported by one or more participant (e.g. PCB157 in Ampoule 1, PCB77 in Ampoule 3, and PCB27 in Ampoule 4).

The range of results among the different labs was frequently quite high, with a %RSD for many of the isomers greater than 20%, emphasizing the "not normal" distribution of the data. Of particular note is the %RSD of 155% for PCB8 in Ampoule 4, which has a bimodal distribution. This suggests poor agreement among the participating laboratories. While the biased high and biased low participants compensate each other when calculating the interlaboratory mean for this interlaboratory study results, problems can occur when merging data sets from these laboratories. To reduce the possibility of laboratories contributing data sets to the central IADN database differing by 20% or more, the use of a common reference standard can minimize this source of variability.

Results for the mono- and di-chlorinated isomers indicated that all of the participants had difficulties with the analysis of these compounds. The results for several of these isomers indicate a bimodal distribution among the participants. For PCB4 Laboratories 9213 (A&B) and 9214 agreed with each other, and Laboratories 9212 (-V & -HP) and 9215 agreed with each other, but differed from the other two labs by an order of magnitude. Laboratory 9216 did not have any of the mono- or di-chlorinated congeners on their target list, except for PCB15, which was not on the target list of the other participants. Therefore no comparison could be made of Laboratory 9216's performance with the other participants for the mono- and di-chlorinated PCB isomers. The range of variability was high, with the %RSD being greater than 50% in several cases. The statistical calculations included in Tables 1-4 for these isomers are not valid from a statistical point of view, and are provided only as a very general indicator of the spread of the data. These PCB's have higher vapour pressures[5] than the more chlorinated isomers, making them more susceptible to vaporization at the high temperature point of the gas chromatograph injection port, with subsequent sample loss. This may be a source of low bias for PCB4 by Laboratories 9213 (A&B) and 9214.

A major source of variation among the laboratories is the problem of co-elution of PCB isomers of various congener groups. Several participants reported results that were a combination of two isomers, such for PCB71 and PCB41 in Tables 2 and 4. For the purposes of calculating an interlaboratory mean, median, and standard deviation, the total value reported for a combination of isomers was divided equally among the isomers and these values were flagged in Tables 1-4. This proportioning of the reported result may not be analytically correct, as the area of the analytical peak may actually be divided 40-60 between the two isomers, or some other proportion. This can introduce biases when evaluating the "accuracy" of the interlaboratory mean of some of the isomers.

The study design attempted to avoid as many co-eluters as possible in Ampoules 1, 2, and 3 and thereby avoid identification errors. However, there were still some problems for some of the participants. As an example, in Ampoule 1, PCB163 was present, but PCB138, a close eluter to PCB163[4], was not included in this ampoule. Two participants reported a positive response for PCB138 and indicated that PCB163 was not part of their target list (Table 1). In Ampoule 4, where both isomers were present together, these two participants reported a value for PCB138 that corresponds to the sum of PCB138 and PCB163 in this ampoule (Table 4). This type of incorrect identification and quantitation due to co-eluters will introduce between-laboratory bias. Further instrumental research and development is required to attempt to eliminate the co-elution problem.

As an alternate way of evaluating the results, a graphical technique was used for those isomers with 6 or 7 results. As each isomer had a "pair" of results, one from either Ampoule 1, 2, or 3, and the other from Ampoule 4, these results may be plotted on an X-Y plot using the Youden technique[6]. The result from Ampoule 1, 2, or 3 is plotted on the vertical axis and the result from Ampoule 4 is plotted on the horizontal axis. The graphs are divided into four quadrants, with the intersection point at the target values. The data points should cluster around the target if random error is the only source of variability. Results in the upper right quadrant are considered biased high and those in the lower left quadrant are biased low. The main source of this type of variability is a difference in analytical standards or inadequate calibration practices. Data points that fall in the lower right or upper left quadrants are considered erratic or out-of-control. Sources of this type of error are more difficult to ascertain. In this study, the participants were analyzing ampoules for direct instrumental injection. Sources of erratic performance could be poor sample injection into the gas chromatograph, a septum leak, poor chromatography if contamination remained from a previous sample, or other instrumental problems. Within-laboratory precision may be assessed by drawing a line between the origin and the intersection of the target values. The closer the data point is to this diagonal line, the better the within-laboratory precision.

The Youden plots for 36 of the PCB isomers in this study are found in Appendix 1, Figures 1 - 36. Isomers such as PCB44 (Fig. 9), PCB49 (Fig. 11), PCB52 (Fig. 12), PCB70 (Fig. 14), PCB101 (Fig. 17), PCB114 (Fig. 19), PCB137 (Fig. 23), PCB118 (Fig.

20), PCB158 (Fig. 27), PCB180 (Fig. 30), PCB187 (Fig. 31), and PCB198 (Fig. 35), demonstrate between-laboratory differences that can usually be attributed to differences in standards. All of these figures demonstrate the spread of the participants' results in the upper right and lower left quadrants along the diagonal line between the origin and the target. As noted above, the use of a common reference standard by all of these laboratories would reduce this type of variation. The participants do demonstrate good within-laboratory precision for these isomers, as the data points are close to the diagonal line between the origin and target for almost all of the laboratories.

The results for PCB4 (Fig. 1) also demonstrates a spread of results in the lower left quadrant. However, as discussed above, the low bias may be due to vaporization at the high temperature point of the GC injection port. Further investigation is required by the participants with low biases to determine if the problems are with their analytical standards or instrumental conditions.

Several isomers demonstrate a pattern of a parallel line to the line passing between the origin and target (PCB 18, Fig. 3, PCB31, Fig. 5, PCB97, Fig. 16, PCB 136, Fig. 22, PCB153, Fig. 25, and PCB158, Fig. 27). These laboratories are precise to this parallel line, suggesting a consistent bias in all of these participants. This may possibly be due to an error in the ratios of concentration to response factors. Only Laboratory 9214 reported two of these isomers as co-eluting with another isomer (PCB31 with PCB28 and PCB153 with PCB105), so a co-elution effect does not appear to be the source of error.

Other isomers such as PCB8 (Fig. 2), PCB33 (Fig. 6), PCB42 (Fig. 8), PCB47 (Fig. 10), PCB66 (Fig. 13), PCB 77 (Fig. 15), PCB128 (Fig. 21), PCB138 (Fig. 24), and PCB190 (Fig. 32) show more erratic performance among the participants. The sources of these types of variation are more difficult to identify, as noted above. Co-elution problems may be the main source of variability for isomers such as PCB8 and PCB138.

Many of the Youden plots re-emphasize the spread in results among the participants, despite the good within-laboratory precision. The interlaboratory mean and median showed agreement with the target for isomers such as PCB18, PCB42, PCB52, PCB101, PCB190, and PCB198. However the plots for these isomers' results (Figures 3, 8, 12, 17, 32, and 35 respectively) demonstrate a wide spread of results among the participants.

INDIVIDUAL LABORATORY PERFORMANCE

To give an overview of the individual participant's agreement with the target values, the results for all of the isomers from all of the ampoules were grouped according to percentage of target. These results are presented in Table 7 and Figure 37 (excluding Laboratory 9211).

Laboratory 9211

Laboratory 9211 reported only total congener groups, and as noted above, they were not included in the interlaboratory performance evaluation. When comparing their results of the total congener groups (based on the number of isomers present in each ampoule), they agree with the target totals within ± 20% for the majority of congener groups, with a few results within 30% of the target (Table 8). The only congener group that they had consistent problems in quantitation was the octachlorobiphenyls.

In all four ampoules they recognized the correct number of isomers present, but their quantification was low compared to the target and the other participants. They should investigate the accuracy of the standard they use for this congener group.

Laboratory 9211 was the only participant to use a Mass Spectrometer as the detection system.

The IADN program requires isomer specific analysis as well as total congener group results[2]. This laboratory will need to develop the capability for isomer-specific analysis to be a future contributor to the IADN program.

Laboratory 9212

This laboratory reported two sets of results, using two different instruments, as described in Table 6. The results in Tables 1-4 have been designated "V" for the Varian 3400 and "HP" for the Hewlett-Packard 5890. Both instruments used the same type of capillary columns and the same calibration standard was used.

The performance of the two systems is not identical. There were several instances of a positive identification on one instrument and not on the other, particularly for the lower concentration ampoules (1, 2, and 3). PCB190, which was identified correctly on the Varian and had good agreement with the target, was not identified on the HP in Ampoule 3, and had a co-elution problem in Ampoule 4. The reported result from the HP in Ampoule 4 was divided equally between PCB170 and PCB190. Based on the non-identification in Ampoule 3 of PCB 190, the correct identification of PCB170 in Ampoule 1, and the value of the combined result in Ampoule 4 corresponding to the target value of only 1 of the 2 isomers (ie. half of the total), the result on the HP for the combined PCB170 and PCB190 probably is attributable to only PCB170.

As seen in Table 7 and displayed in Figure 37, the distribution of sample concentrations on the Varian and HP are different in the ranges 75-90% and 90-110%. Using the Varian, 54.7% and 33.7% of the results were in the ranges 75-90% and 90-100% respectively, as compared to using the HP where 19.8% and 46.5% were in the 75-90% and 90-110% range of target. As noted above, the same calibration standard was used for both analytical systems. The differences between the two sets of results appear to be instrument related, possibly due to differences in temperature programming, gas flow rates, detectors, calibration factors or integration software. Laboratory 9212 should investigate further the differences in performance of the two instrumental systems so that they can report a consistent data set using either analytical system.

The within-laboratory precision is generally consistent (see Figures 1 - 36) in Appendix 1, though there are some instances of missed results in the "low" ampoules (1, 2, and 3). As detection limits were not provided, some of the "low" concentration levels may have been near the detection limits for some of the isomers.

Laboratory 9213

Laboratory 9213 analyzed the ampoules using a Varian 3400 with a SPI (Septum Programmable Injector). They repeated their analysis of the PCB ampoules using the Varian Vista 6000. Both systems used the same capillary column and the same calibration standard was used. Results from the Varian 3400 are labelled "A" and results from the Varian 6000 are labelled "B".

The results from the two systems are not identical. As seen in Table 7 and Figure 37, 44.6% of the results from the Varian 3400 ("A") are within 90-110% of the target, while only 20.7% of the results from the Varian 6000 ("B") are in the same range. The majority of results from the Varian 6000 are in the range 75-90% of target (Table 7).

Within-laboratory precision was good for many of the isomers. However there was inconsistent performance for some of the isomers, such as PCB33 (Fig. 6), PCB47 (Fig. 10), PCB 128 (Fig. 21), and PCB177 (Fig. 29). The Varian 3400 performed slightly better than the Varian 6000 in this regard.

As with Laboratory 9212, who also reported results from two different analytical systems, Laboratory 9213 used the same calibration standard on both systems. As noted for Laboratory 9212, differences in temperature programming, gas flow rates, detectors or integration software may be potential sources of variability between the two systems. Further investigation of the differences in performance of the two instrumental systems should be conducted so that this laboratory can report a consistent data set using either analytical system.

Laboratory 9214

Laboratory 9214 had the highest proportion of results that were greater than the target value as well as the results of other participants (Table 7 and Figure 37). Evaluation of their standard to a reference standard should help to resolve these differences. They also had problems with the analysis of the mono- and di-chlorobiphenyls. Several results are either very low (eg. PCB4 in Ampoules 2 and 4) or high (eg. PCB5 in Ampoule 3). Better performance (i.e. agreement with the target) was achieved on the more highly chlorinated isomer groups. Investigation of their initial GC conditions may help improve performance for the lower chlorinated groups which elute near the beginning of the GC scan.

They also had the largest number of co-eluters, as compared to the other participants. While they were not the only laboratory to use a 30 m capillary column (Table 6), switching to a 60 m column may help eliminate some of the co-elution problems.

Within-laboratory precision is very good for almost all of the isomers (Figures 1 - 36), despite some of the other problems noted above. Isomers which had less precise within-laboratory performance generally were co-eluters such as PCB105 (Fig. 18) and PCB153 (Fig. 25).

Laboratory 9215

Laboratory 9215 had 39.7% of its results within 90-110% of the target. They had no co-elution problems with Ampoules 1, 2, and 3, but they had some interference problems for PCB17 and PCB149 in Ampoule 4. They demonstrated good within-laboratory precision for all the isomers (Figures 1 - 36) except PCB18 (Fig. 3) and PCB194 (Fig. 33).

Laboratory 9216

Laboratory 9216 had run out of their own calibration standards when the ampoules were received. To try and meet the study deadline, they borrowed calibration standards that had been cross-checked to the same external sources that they used.

When they received the table of results from this study, they felt that their performance could have been better. Upon comparing the borrowed standards with their own new standards, Laboratory 9216 felt that the borrowed standards were the source of bias in their performance. They reanalysed the ampouled solutions using their new calibration standards. Both sets of results are included in Tables 1-4, but the revised results are used in the calculations of interlaboratory mean, median and standard deviation. The following discussion of their performance refers to the revised set of results.

Laboratory 9216 had 48% of their results within 90-110% of the target (Table 7 and Figure 37). They tended to be biased high, as 30% of their results were in the range 110-130% of the target. They had the fewest number of interlaboratory study PCB isomers on their target list, as compared with the other participants, though they provided a list of the different isomers that are on their target list. They particularly didn't have many of the mono-, di-, and tri-chlorobiphenyls on their own target list.

Within-laboratory precision was variable. For some isomers (eg. PCB44 (Fig. 9), PCB101 (Fig. 17), PCB105 (Fig. 18), PCB114 (Fig. 19), PCB187 (Fig. 31), and PCB209 (Fig. 36)), they had excellent within-laboratory precision. For others (eg. PCB31 (Fig. 5), PCB40 (Fig. 7), PCB77 (Fig. 15), and PCB138 (Fig. 24)) they had more erratic performance. It is difficult to determine the possible sources of this variability, as the different isomers were all part of a combined solution. More of the variability appears to be in Ampoule 4, which contained all of the isomers in the study target list. While Laboratory 9216 did not note any co-elution problems, integration of GC peaks of closely eluting isomers may have been more difficult, contributing to the increased variability of the results from Ampoule 4.

5 CONCLUSION

The results from this study demonstrate an interlaboratory mean and median that appear to agree with the target for PCB isomers that have three or more chlorine atoms. However, the between-laboratory variability is frequently at a range of 20% or greater, indicating a spread of results that suggest poor agreement among the participating laboratories. This may introduce greater biases to the IADN database than may be acceptable. The use of a common reference standard by the participants should reduce this source of variability.

The participants demonstrated problems with the analysis of the mono- and di-chlorinated biphenyls. As these are more volatile compounds, sample losses may have occurred at the GC injection port. Differences in standards may also contribute to between-laboratory variability.

Co-elution of PCB isomers also contributes to the between-laboratory variability and affects the accuracy of the results. The participants were not asked to provide copies of their chromatograms nor details of their analytical conditions (i.e. retention times, resolution, etc.), so the degree of co-elution contributing to between-laboratory variability cannot be determined for this study. Future studies may attempt to address the degree of this effect in greater detail. As technology improves, resolution of different isomer pairs should help reduce between-laboratory variability.

At the time of this study, the IADN Implementation Committee had not selected a final list of PCB isomers to be the target list for IADN. The broad range of isomers used in the study was intended to cover all the possibilities. When a final parameter list is chosen, future studies will focus on those PCB isomers so that more definitive performance criteria may be developed.

6 REFERENCES

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7 APPENDIX 1 - RESULTS AND GRAPHS

Table 1	Ampoule PCBs 1, Results in μg/L
Table 2	Ampoule PCBs 2, Results in μg/L
Table 3	Ampoule PCBs 3, Results in μg/L
Table 4	Ampoule PCBs 4, Results in μg/L
Table 5	Distribution of PCB Isomers on Participants' Target Lists
Table 6	Instruments and GC Columns
Table 7	Distribution of Participants' Results Relative to Target
Table 8	Performance of Laboratory 9211
Figures 1 - 36	Youden Plots
Figure 37	Distribution of Participants' Results Relative to Target

TABLE 1 - Ampoule PCBs 1
Results in μg/L

PCB ISOMER	TARGET	9211	#	9212-V	9212-HP	9213A	9213B	9214	9215	9216	MEAN	MEDIAN	SD	n	%RSD	9216 $
PCB3	232			N/A	N/A	234.91	155.92	166.408	227.01	N/A	196.1	196.71	40.65	4	20.73%	N/A
TOTAL MONO	232	210	1	-	-	234.91	155.92	166.408	227.01	-						-
PCB4/10								0.081								
PCB5						16.31		40.559	16.59							
PCB7	300			N/A	N/A	379.66	167.48	123.851	283.52	N/A	238.6	225.5	115.7	4	48.47%	N/A
PCB16	11.3			N/A	N/A	10.84	8.86	7.598	11.43	N/A	9.682	9.85	1.772	4	18.30%	N/A
TOTAL DI	311.3	260	2	-	-	406.81	176.34	172.089	311.54	-						-
PCB18	10.4			9.8	10.7	8.66	6.65	6.861	12.62	12.3	9.654	9.8	2.406	7	24.93%	19.68
PCB19	10.4			N/A	N/A	8.53	7.61	5.912	10.93	N/A	8.496	8.57	2.195	4	25.84%	N/A
PCB24	9.4			N/A	N/A	10.78	13.13 &	7.955 &	6.26	N/A	9.531	9.37	3.038	4	31.88%	N/A
PCB29	16.4			14.0	14.8	N/A	N/A	12.351	15.30	N/A	14.11	14.4	1.291	4	9.15%	N/A
PCB33						8.67										
TOTAL TRI	46.6	41	5	23.8	25.5	37.63	27.39	33.079	45.11	12.3						19.68
PCB40	10.8			12.8	10.9	12.47	10.67	11.737	11.17	10.8	11.51	11.17	0.85	7	7.39%	16.53
PCB44	11.0			10.3	11.2	9.69	8.47	11.484	11.11	10.9	10.45	10.9	1.064	7	10.18%	16.61
PCB48	10.1			N/A	N/A	9.14	7.74	9.671	8.93	N/A	8.87	9.04	0.815	4	9.19%	N/A
PCB52	9.9			10.1	10.2	9.33	7.58	8.058	10.51	12.0	9.683	10.1	1.51	7	15.60%	17.51
PCB53	10.5			0.0	0.0	N/A	N/A	9.135	8.08	N/A	4.304	4.04	4.988	4	115.90%	N/A
PCB56/60						0.22										
PCB70	13.5			0.0	14.2	14.80	11.87	17.752	14.04	N/A	12.11	14.12	6.227	6	51.42%	N/A
PCB77								0.253								
PCB81	9.7			N/A	N/A	N/A	N/A	6.932	9.95	N/A	8.441	8.44	2.134	2	25.28%	N/A
TOTAL TETRA	75.5	78	7	33.2	46.5	55.65	46.33	75.022	73.79	33.7	-					50.65
PCB84/92								0.046								
PCB87							5.11									
PCB95								0.030								
PCB97	12.8			12.0	13.0	13.91	ND	11.024	10.11	N/A	10.01	11.51	5.086	6	50.82%	N/A
PCB105	9.5			7.7	9.2	8.39	6.50	9.488	8.77	9.4	8.493	8.77	1.08	7	12.72%	10.67
PCB114						0.21		0.093								
PCB118	10.1			8.5	10.2	10.90	8.41	10.200	9.92	ND	8.304	9.92	3.776	7	45.47%	ND
PCB126	12.0			N/A	N/A	N/A	N/A	N/A	N/A	N/A	-					N/A
TOTAL PENTA	44.4	34	4	28.2	32.4	33.41	20.02	30.881	28.80	9.4	-					10.67
PCB129/178							6.08 $									
PCB130/176						1.01 $										
PCB136	20.0			15.8	17.2	20.15	17.77	12.087	18.71	N/A	16.95	17.49	2.794	6	16.48%	N/A
PCB137/176								0.234								
PCB138						11.23	11.10			11.25						13.01
PCB146						0.20										
PCB149	12.2			N/A	N/A	11.46	10.30	11.270	12.86	N/A	11.47	11.37	1.055	4	9.20%	N/A
PCB157	14.8			0.0	0.0	16.54	12.99	0.000	N/A	N/A	5.906	0.0	8.184	5	138.57%	N/A

TABLE 1 - Ampoule PCBs 1
Results in µg/L

PCB ISOMER	TARGET	9211	#	9212-V	9212-HP	9213A	9213B	9214	9215	9216	MEAN	MEDIAN	SD	n	%RSD	9216 §
PCB163	10.8			N/A	N/A	N/A	N/A	11.654 *	12.91	N/A	12.28	_	0.888	2	7.23%	N/A
PCB166	21.9			N/A	N/A	N/A	N/A	20.608	N/A	N/A						N/A
TOTAL HEXA	79.7	81	5	15.8	17.2	60.59	58.24	55.853	44.48	11.25	-					13.01
PCB170	13.3			11.5	11.6	11.83	10.43	22.470 @	11.86	14.5	13.46	11.83	4.163	7	30.94%	15.26
PCB176/130						1.01 $										
PCB177				0.2		0.07		0.138								
PCB178/129							6.08 $									
PCB180								0.036								
PCB187/182								0.070								
PCB189							0.14	0.126								
TOTAL HEPTA	13.3	6.9	1	11.7	11.6	12.91	16.65	22.841	11.86	14.5	-					15.26
PCB194	11.0			9.2	11.8	9.55	10.70	10.307	12.08	11.4	10.72	10.70	1.105	7	10.31%	11.25
TOTAL OCTA	11.0	6.6	1	9.2	11.8	9.55	10.70	10.307	12.08	11.4	-					11.25

#	Laboratory 9211 reported only total congener groups. The values in the adjacent column indicate the total number of analytical peaks detected by this laboratory.
&, @, *	These isomers were reported as co-eluting with another isomer. In this ampoule, the co-eluting isomer was not present, so the value reported may be attributed to the one isomer only.
$	These isomers were reported as co-eluting with another isomer. The result is equally divided among both isomers.
N/A	Laboratory does not calibrate for this isomer.
ND	Participant did not detect this isomer, which was present in the ampoule, and reported as Not Detected.
	Participants that did not report a result for an isomer that was present in the ampoule, and did not mark the report form with an "ND", were assigned a "0".
§	Original set of results reported by Laboratory 9216. They had run out of their own calibration standards when the ampoules were received. To try and meet the study deadline, they borrowed calibration standards that had been cross-checked to the same external sources that they used. When they received the table of results from this study, they did felt that their performance could have been better. Upon comparing the borrowed standards with their own new standards, Laboratory 9216 felt that the borrowed standards were the source of variability in their performance. They reanalysed the ampouled solutions using their new calibration standards. The revised results are used in the calculations of mean and standard deviation.

Laboratories 9212 and 9213 provided two sets of results using two different instruments. See Table 8 for description.

TABLE 2 - Ampoule PCBs 2
Results in μg/L

PCB ISOMER	TARGET	9211	#	9212-V	9212-HP	9213A	9213B	9214	9215	9216	MEAN	MEDIAN	SD	n	%RSD	9216 $
PCB4	246			208.6	205.6	27.29	20.29	28.103	188.37	N/A	113.0	108.2	96.48	6	85.35%	N/A
PCB7							3.02									
PCB8	228			226.1	227.4	147.83	112.97	363.986	230.18	N/A	218.1	226.8	86.68	6	39.75%	N/A
TOTAL DI	474	320	2	434.7	433.0	175.12	136.28	392.089	418.55	-	-					-
PCB16						14.50										
PCB17	9.9			N/A	N/A	N/A	9.54	9.961	7.06	N/A	8.854	9.54	1.568	3	17.71%	N/A
PCB25	10.8			N/A	N/A	10.92	9.80	9.872	9.45	N/A	10.01	9.84	0.634	4	6.33%	N/A
PCB28	14.5			11.4	0.0	12.24	10.92	15.426 @	11.68	N/A	10.28	11.54	5.284	6	51.42%	N/A
PCB31							0.29									
PCB32	10.4			N/A	N/A	N/A	12.61 ▸	21.405	11.58	N/A	15.2	12.61	5.4	3	35.53%	N/A
PCB33	10.3			0.0	9.0	10.95	9.84	11.737	10.28	N/A	8.635	10.06	4.332	6	50.17%	N/A
TOTAL TRI	55.9	43	5	11.4	9.0	48.61	53	68 401	50.05	-	-					-
PCB40							0.73			3.41						
PCB41	10.4			N/A	N/A	6.215 $	5.60 $	16.154 $	4.48 $	N/A	8.112	5.91	5.409	4	66.68%	N/A
PCB44							0.68									
PCB47						15.31	11.82	10.829								
PCB49	9.9			7.7	9.2	9.36	7.41	8.623	8.43	11.0	8.818	8.62	1.199	7	13.59%	18.18
PCB52							0.43	0.040								
PCB56/60						0.24				2.1						
PCB61	10.6			10.6	9.8	N/A	N/A	N/A	N/A	N/A	10.2	-	0.566	2	5.55%	N/A
PCB64						19.07	14.09									
PCB71	11.1			N/A	N/A	6.215 $	5.60 $	16.154 $	4 48 $	N/A	8.112	5.91	5.409	4	66.68%	N/A
PCB74						15.92										
PCB75	10.4			12.3	12.1	N/A	N/A	N/A	N/A	N/A	12.2	-	0.141	2	1.16%	N/A
TOTAL TETRA	52.4	60	4	30.6	31.1	72.33	46.36	51.800	26.35	16.51	-					18.18
PCB84/92								0.584								
PCB95	13.3			N/A	N/A	13.25	10.62	14.142	14.59	N/A	13.15	13.70	1.777	4	13.51%	N/A
PCB97							0.7	0.080								
PCB100	11.4			N/A	N/A	N/A	N/A	15.765	11.87	N/A	13.82	-	2.754	2	19.93%	N/A
PCB101							0.56	0.144								
PCB110	11.0			N/A	N/A	7.84	6.18	9.039	8.87	N/A	7.982	8.36	1.313	4	16.45%	N/A
PCB119	12.4			N/A	N/A	N/A	N/A	14.286	12.19	N/A	13.24		1.482	2	11.20%	N/A
TOTAL PENTA	48.1	38	4	-	-	21.09	18.06	54.040	47.52	-	-					-
PCB128	16.7			15.4	18.2	14.06	12.66	27.188	16.95	17.2	17.38	16.95	4.731	7	27.22%	21.56
PCB130/176						1.96										
PCB132							1.37									
PCB136							0.96									
PCB138	9.9			8.5	0.0	9.17	9.20	10.861 •	6.18	11.2	7.873	9.17	3.845	7	48.83%	12.65
PCB149							1.03	0.037								
PCB155	10.9			N/A	N/A	N/A	N/A	N/A	N/A	N/A						N/A

TABLE 2 - Ampoule PCBs 2
Results in µg/L

PCB ISOMER	TARGET	9211	#	9212-V	9212-HP	9213A	9213B	9214	9215	9216	MEAN	MEDIAN	SD	n	%RSD	9216 $
PCB157/201							0.81									
PCB167								0.082								
PCB169	9.5			N/A	N/A	N/A	N/A	N/A	7.87	N/A						N/A
TOTAL HEXA	47.0	42	4	23.9	18.2	25.19	26.03	38.168	31.00	43.6	-					34 21
PCB175						0.08	1.13									
PCB177	15.1			14.9	17.0	14.53	16.54	17.011	16.61	N/A	16.10	16.58	1.095	6	6.80%	N/A
PCB187/182							0.31	0.054								
PCB189	11.1			N/A	N/A	11.89	ND	9.829	11.55	12.8	9.214	11.55	5.262	5	57.11%	13.45
TOTAL HEPTA	26.2	20	2	14.9	17.0	0.19	17.98	26.894	28.16	12.8	-					13.45
PCB194						26.69	0.61									
PCB198	13.9			11.8	13.8	13.78	15.30	18.235	8.20	N/A	13.52	13.79	3.371	6	24.93%	N/A
TOTAL OCTA	13.9	6	1	11.8	13.8	40.47	15.91	18.235	8.20	-						
PCB209	11.8			9.2	11.6	10.39	10.09	14.528	10.58	11.6	11.14	10.58	1.715	7	15.39%	11.00
TOTAL DECA	11.8	13	1	9.2	11.6	10.39	10.09	14.528	10.58	11.6	-					11.00

# Laboratory 9211 reported only total congener groups. The values in the adjacent column indicate the total number of analytical peaks detected by this laboratory.

&, @, •, ▸ These isomers were reported as co-eluting with another isomer. In this ampoule, the co-eluting isomer was not present, so the value reported may be attributed to the one isomer only.

$ These isomers were reported as co-eluting with another isomer. The result is equally divided among both isomers.

N/A Laboratory does not calibrate for this isomer.

ND Participant did not detect this isomer, which was present in the ampoule, and reported as Not Detected.

Participants that did not report a result for an isomer that was present in the ampoule, and did not mark the report form with an "ND", were assigned a "0".

$ Original set of results reported by Laboratory 9216. They had run out of their own calibration standards when the ampoules were received. To try and meet the study deadline, they borrowed calibration standards that had been cross-checked to the same external sources that they used. When they received the table of results from this study, they did felt that their performance could have been better. Upon comparing the borrowed standards with their own new standards, Laboratory 9216 felt that the borrowed standards were the source of variability in their performance. They reanalysed the ampouled solutions using their new calibration standards. The revised results are used in the calculations of mean and standard deviation.

Laboratories 9212 and 9213 provided two sets of results using two different instruments. See Table 8 for description.

TABLE 3 - Ampoule PCBs 3
Results in μg/L

PCB ISOMER	TARGET	9211	#	9212-V	9212-HP	9213A	9213B	9214	9215	9216	MEAN	MEDIAN	SD	n	%RSD	9216 $
PCB3				N/A	N/A			1.041								
TOTAL MONO								1.041								
PCB4/10							1.24	0.089								
PCB5	234			N/A	N/A	244.76 $	218.36 $	560.844	214.33	N/A	309.6	231.6	168.1	4	54.29%	N/A
PCB7						0.29	0.65	0.249								
PCB15	198			N/A	N/A	N/A	N/A	N/A	N/A	283.0						331.3
TOTAL DI	432	360	2	-	-	245.05	220.25	561.182	214.33	283.0	-					331.3
PCB16							0.64	0.164								
PCB17							32.10									
PCB22	13.4			N/A	N/A	13.47	11.55	20.676	11.91	N/A	14.4	12.69	4.265	4	29.62%	N/A
PCB24						10.53										
PCB25							0.61	0.206								
PCB26	10.6			N/A	N/A	12.92	11.82	9.795	12.94	N/A	11.87	12.37	1.478	4	12.45%	N/A
PCB27	12.8			N/A	N/A	ND	12.61 ▸	8.841 &	14.09	N/A	8.885	10.73	6.322	4	71.15%	N/A
PCB31	12.1			10.6	11.6	11.27	9.85	11.500 @	11.16	12.6	11.23	11.27	0.856	7	7.62%	19.78
PCB33						0.54	1.84	0.225								
PCB37	9.5			N/A	N/A	N/A	N/A	7.020	13.48	N/A	10.25	-	4.568	2	44.56%	N/A
TOTAL TRI	58.4	50	5	10.6	11.6	48.73	81.02	58.427	63.58	12.6	-					19.78
PCB40				4.1	4.0	4.21	3.90	3.682								
PCB41/71							0.48	0.074								
PCB42	13.9			13.2	15.4	16.92	14.15	13.147	12.74	N/A	14.26	13.68	1.616	6	11.33%	N/A
PCB47	13.7			11.2	11.6	16.02	11.10	14.168	11.80	N/A	12.65	11.7	2.000	6	15.81%	N/A
PCB49							0.33	0.057								
PCB52								0.027								
PCB56/60						1.71	2.51									
PCB64							0.94									
PCB66	14.8			10.5	12.8	ND	11.44	20.916	16.41	N/A	12.01	12.12	7.016	6	58.42%	N/A
PCB70/76								0.059								
PCB74	10.5			N/A	N/A	9.95	10.98	8.990	14.09	N/A	11.00	10.47	2.213	4	20.11%	N/A
PCB77	10.6			0.0	9.6	ND	ND	11.904	13.27	15.0	7.111	9.6	6.844	7	96.25%	19.35
TOTAL TETRA	63.5	79	6	39.0	53.4	48.81	55.83	73.024	68.31	15.0	-					19.35
PCB84	13.0			10.4	11.4	0	0	20.448	15.03	N/A	9.546	10.9	8.19	6	85.79%	N/A
PCB95						16.72										
PCB100								0.531								
PCB101	10.5			8.7	9.4	11.00	20.06	8.480	11.83	12.2	11.67	11.00	3.985	7	34.16%	13.95
PCB110							4.66	0.258								
PCB114	13.1			13.1	12.0	14.05	14.10	0.000	15.31	12.5	11.58	13.1	5.225	7	45.12%	15.02
PCB118								0.197								
PCB119								0.028								
PCB123	11.4			N/A	N/A	N/A	N/A	N/A	N/A	N/A						N/A

TABLE 3 - Ampoule PCBs 3
Results in μg/L

PCB ISOMER	TARGET	9211	#	9212-V	9212-HP	9213A	9213B	9214	9215	9216	MEAN	MEDIAN	SD	n	%RSD	9216 $
TOTAL PENTA	48.0	40	4	32.2	32.8	41.77	38.82	29.942	42.17	24.7	-					28.97
PCB128						10.88	13.50	0.132								
PCB132							1.36									
PCB137	13.1			11.8	12.6	14.71	16.74	0.000	8.57	12.9	10.9	12.6	5.321	7	48.80%	16.7
PCB138/163						0.19		0.096								
PCB146						13.90										
PCB149							15.36									
PCB151						0.23										
PCB153	19.5			15.8	16.8	18.92	16.05	23.232	17.73	19.8	18.33	17.73	2.609	7	14.23%	25.22
PCB156	14.2			0.0	11.5	13.41	14.55	0.000	15.42	15.2	10.01	13.41	6.964	7	69.56%	16.55
PCB157/201							0.10									
PCB158	9.6			8.4	8.5	9.57	11.66	16.727	12.26	N/A	11.19	10.62	3.152	6	28.18%	N/A
PCB167	12.8			10.1	13.1	N/A	N/A	11.687	15.94	N/A	12.71	12.40	2.479	4	19.51%	N/A
TOTAL HEXA	69.2	72	5	46.1	62.5	81.81	88.32	51.874	69.92	47.9	-					58.47
PCB177							1.29	0.066								
PCB180	11.7			10.0	10.8	10.73	9.25	12.567	8.80	12.9	10.72	10.73	1.556	7	14.52%	13.33
PCB187	13.9			12.0	13.3	13.45	12.08	13.957	11.63	14.6	13	13.3	1.117	7	8.59%	15.97
PCB189							0.99									
PCB190	10.0			9.5	0.0	9.74	12.55	15.843	10.73	N/A	9.727	10.24	5.311	6	54.60%	N/A
TOTAL HEPTA	35.6	30	3	31.5	24.1	33.92	36.16	42.433	31.16	27.5	-					29.3
PCB195	12.7			11.2	11.5	11.70	10.56	29.862	14.18	12.3	14.47	11.70	6.882	7	47.56%	13.23
PCB198							0.85									
TOTAL OCTA	12.7	6.2	1	11.2	11.5	11.70	11.41	29.862	14.18	12.3	-					13.23
PCB209							0.44									

#	Laboratory 9211 reported only total congener groups. The values in the adjacent column indicate the total number of analytical peaks detected by this laboratory.
$, &, @, ▸	These isomers were reported as co-eluting with another isomer. In this ampoule, the co-eluting isomer was not present, so the value reported may be attributed to the one isomer only.
N/A	Laboratory does not calibrate for this isomer.
ND	Participant did not detect this isomer, which was present in the ampoule, and reported as Not Detected. Participants that did not report a result for an isomer that was present in the ampoule, and did not mark the report form with an "ND", were assigned a "0".
$	Original set of results reported by Laboratory 9216. They had run out of their own calibration standards when the ampoules were received. To try and meet the study deadline, they borrowed calibration standards that had been cross-checked to the same external sources that they used. When they received the table of results from this study, they did felt that their performance could have been better. Upon comparing the borrowed standards with their own new standards, Laboratory 9216 felt that the borrowed standards were the source of variability in their performance. They reanalysed the ampouled solutions using their new calibration standards. The revised results are used in the calculations of mean and standard deviation.

Laboratories 9212 and 9213 provided two sets of results using two different instruments. See Table 6 for description.

TABLE 4 - Ampoule PCBs 4
Results in μg/L

PCB ISOMER	TARGET	9211	#	9212-V	9212-HP	9213A	9213B	9214	9216	9216	MEAN	MEDIAN	SD	n	%RSD	9216 $
PCB3	348			N/A	N/A	360.79	250.30	231.156	298.44	N/A	285.2	274.4	57.82	4	20.27%	N/A
TOTAL MONO	348	330	1			360.79	250.30	231.156	298.44	-	-					-
PCB4	369			292.4	259.2	37.80	29.05	36.256	265.26	N/A	153.3	148.5	130.8	6	85.32%	N/A
PCB5	351			N/A	N/A	657.35	73.26 +	625.532 +	352.89	N/A	427.25	439.2	222.7	4	63.83%	N/A
PCB7	450			N/A	N/A	642.58	239.56	175.314	378.67	N/A	359.0	309.12	207.2	4	57.71%	N/A
PCB8	342			0.0	0.0	0.00	73.26 +	625.532 +	267.98	N/A	161.12	36.3 ••	250.1	6	155.20%	N/A
PCB15	297			N/A	N/A	N/A	N/A	N/A	N/A	440.0						654.1
TOTAL DI	1809	1490	4	292.4	259.2	1337.73	415.13	1462.634	1264.80	440.0	-					654.1
PCB16	22.6			N/A	N/A	43.64	18.25 ▾	14.045	22.21	N/A	24.54	20.23	13.16	4	53.66%	N/A
PCB17	19.8			N/A	N/A	N/A	56.75	78.544	INT	N/A	67.65	-	15.41	2	22.78%	N/A
PCB18	20.8			16.8	19.3	15.87	12.64	10.948	29.94	20.8	18.04	16.8	6.279	7	34.80%	27.1
PCB19	20.8			N/A	N/A	17.88	14.58	11.156	19.67	N/A	15.82	16.23	3.758	4	23.75%	N/A
PCB22	26.8			N/A	N/A	24.99	20.20	41.913	23.19	N/A	27.57	24.09	9.762	4	35.40%	N/A
PCB24	18.8			N/A	N/A	35.94	27.84 «	16.997	12.27	N/A	23.26	22.42	10.67	4	45.88%	N/A
PCB25	21.6			N/A	N/A	21.81	18.01	20.290	19.78	N/A	19.97	20.04	1.567	4	7.85%	N/A
PCB26	21.2			N/A	N/A	23.75	20.84	19.487	25.74	N/A	22.45	22.30	2.822	4	12.57%	N/A
PCB27	25.6			N/A	N/A	ND	27.86 «	16.997 &	25.37	N/A	17.56	21.18	12.59	4	71.73%	N/A
PCB28	29.0			23.5	24.1	24.66	19.38	28.256 @	24.77	N/A	24.11	24.38	2.852	6	11.83%	N/A
PCB29	32.8			25.4	27.6	N/A	N/A	25.292	28.53	N/A	26.71	26.5	1.616	4	6.05%	N/A
PCB31	24.2			19.3	19.1	17.22	16.83	28.256 @	21.06	ND	17.4	19.1	8.572	7	49.28%	ND
PCB32	20.8			N/A	N/A	N/A	18.25 ▾	43.837	18.72	N/A	26.94	18.72	14.64	3	54.35%	N/A
PCB33	20.6			26.8	17.1	35.65	29.44	23.205	21.36	N/A	25.59	25.00	6.529	6	25.51%	N/A
PCB37	19.0			N/A	N/A	N/A	N/A	11.180	22.49	N/A	16.84	-	7.997	2	47.50%	N/A
TOTAL TRI	344.4	270	12	111.8	107.2	261.41	300.87	390.404	315.10	20.8	-					27.1
PCB40	21.6			25.0	22.3	27.09	23.97	29.028	24.71	29.3	25.91	25.0	2.635	7	10.17%	35.98
PCB41	20.8			N/A	N/A	12.87 $	10.15 $	32.511 $	8.90 $	N/A	16.11	11.51	11.06	4	68.66%	N/A
PCB42	27.8			25.8	21.0	32.47	24.89	28.580	25.05	N/A	26.3	25.43	3.877	6	14.74%	N/A
PCB44	22.0			18.6	20.8	17.77	15.70	22.978	20.97	22.7	19.93	20.8	2.681	7	13.45%	28.81
PCB47	27.4			24.5	0.0	70.27	47.38	35.999 ▸	23.43	N/A	33.6	30.25	23.9	6	71.13%	N/A
PCB48	20.2			N/A	N/A	0.00	0.00	35.999 ▸	17.42	N/A	13.35	8.71	17.19	4	128.68%	N/A
PCB49	19.8			15.0	16.0	17.22	13.73	16.990	17.04	22.2	16.88	16.99	2.667	7	15.80%	29.46
PCB52	19.8			19.0	19.8	17.34	14.35	15.700	18.70	25.0	18.56	18.70	3.431	7	18.49%	30.63
PCB53	21.0			0.0	0.0	N/A	N/A	17.254	14.96	N/A	8.054	7.48	9.346	4	116.05%	N/A
PCB56/60						3.58										
PCB61	21.2			13.9	20.2	N/A	N/A	N/A	N/A	N/A	17.05	-	4.455	2	26.13%	N/A
PCB64						34.19	25.96									
PCB66	29.6			36.8	46.6	0.00	0.00	42.651	28.08	N/A	25.69	32.44	20.85	6	81.17%	N/A
PCB70	27.0			0.0	25.3	28.40	22.47	35.787	27.69	N/A	23.27	26.50	12.24	6	52.58%	N/A
PCB71	22.2			N/A	N/A	12.87 $	10.14 $	32.511 $	8.90 $	N/A	16.11	11.51	11.06	4	68.69%	N/A
PCB74	21.0			N/A	N/A	56.92	15.78	17.788	26.61	N/A	29.27	22.20	19.02	4	64.98%	N/A

TABLE 4 - Ampoule PCBs 4
Results in μg/L

PCB ISOMER	TARGET	9211	#	9212-V	9212-HP	9213A	9213B	9214	9215	9216	MEAN	MEDIAN	SD	n	%RSD	9216 $
PCB75	20.8			17.4	20.9	N/A	N/A	N/A	N/A	N/A	19.15	-	2.475	2	12.92%	N/A
PCB77	21.2			13.8	16.8	ND	ND	23.940	25.70	68.7	21.28	16.8	23.3	7	109.50%	100.8
PCB81	19.4			N/A	N/A	N/A	N/A	13.603	22.59	N/A	18.1	-	6.355	2	35.12%	N/A
TOTAL TETRA	382.8	440	13	209.8	229.7	330.19	224.52	401.320	328.53	167.9	-					225.7
PCB84	26.0			20.0	20.1	24.19	5.12	42.226	28.43	N/A	23.34	22.15	12.14	6	52.01%	N/A
PCB87							9.89									
PCB95	26.6			N/A	N/A	68.22	47.73	27.469	29.03	N/A	43.11	38.38	19.1	4	44.31%	N/A
PCB97	25.6			22.4	23.4	25.07	22.87	22.243	16.00	N/A	22	22 64	3.11	6	14.14%	N/A
PCB100	22.8			N/A	N/A	N/A	N/A	31.933	23.85	N/A	27.89	-	5.716	2	20.49%	N/A
PCB101	21.0			17.1	18.1	20.90	35.11	17.310	23.05	26.2	22.54	20.90	6.468	7	28.70%	28.03
PCB105	19.0			16.0	17.8	16.32	11.70	32 591 ◂	16.36	19.4	18.6	16.36	6.603	7	35.51%	21.52
PCB110	22.0			N/A	N/A	26.17	16.75	18.357	15.04	N/A	19.08	17.55	4 917	4	25.77%	N/A
PCB114	26.2			25.5	25.5	26.29	25.17	0.141	29 54	28.2	22.91	25.5	10.17	7	44.39%	27.59
PCB118	20.2			18.1	20.2	21.12	15.56	20.341	17.78	ND	16.16	18 1	7.374	7	45.64%	ND
PCB119	24.8			N/A	N/A	N/A	N/A	29 189	22.84	N/A	26.01	-	4.489	2	17.26%	N/A
PCB123	22.8			N/A	N/A	N/A	N/A	N/A	N/A	N/A						N/A
PCB126	24.0			N/A	N/A	N/A	N/A	N/A	N/A	N/A						N/A
TOTAL PENTA	281	220	12	119 1	125 1	228 28	189.9	274.391	221.92	73.8	-					77.14
PCB128	33.4			28 6	33.2	58.41	42.40	57.328	32.46	39.4	41.69	39 4	11.96	7	28 69%	39 68
PCB129/178							11.32 •									
PCB135/144						0.27										
PCB136	40.0			26.2	28.4	36.66	33.16	23.373	32.78	N/A	30.1	30.59	4 958	6	16 48%	N/A
PCB137	26.2			22.2	24.4	25.75	27.19	0.000	16.09	27.6	20 46	24.4	9.836	7	48.07%	27.51
PCB138	19.8			0.0	34.4	48.34	32.00	22.524 •	12.12 •	45.8	27.88	32.00	17.58	7	63.04%	45 6
PCB146						1.04										
PCB149	24.4			N/A	N/A	41.66	42.37	21.739	INT	N/A	35.26	41.66	11.71	3	33.22%	N/A
PCB151						0.60										
PCB153	39.0			29.0	31.6	35.29	27.82	32.591 ◂	33.34	40.7	32.91	32.591	4.273	7	12.99%	45.63
PCB155	21.8			N/A	N/A	N/A	N/A	N/A	N/A	N/A						N/A
PCB156	28.4			25.2	23.2	32.64	25.72	0.216	29.21	32.2	24.06	25.72	11.1	7	46.16%	31.34
PCB157	29.6			24 8	23.4	32.34	25.29	0.000	N/A	N/A	21.17	24.8	12 33	5	58.25%	N/A
PCB158	19.2			17.0	16.4	18.55	20.25	32.621	22.80	N/A	21 27	19 4	6.026	6	28.33%	N/A
PCB163	21.6			N/A	N/A	N/A	N/A	22.524 •	12 12 •	N/A	17.31	-	7 36	2	42.52%	N/A
PCB166	43.8			N/A	N/A	N/A	N/A	43.782	N/A	N/A						N/A
PCB167	25.6			19.6	24.2	N/A	N/A	23.597	32.62	N/A	25	23.90	5 472	4	21.88%	N/A
PCB169	19.0			N/A	N/A	N/A	N/A	N/A	16.03	N/A						N/A
TOTAL HEXA	391.8	380	11	192.6	239.2	331.55	287.52	247.704	253.79	185.7	-					189 8
PCB170	26.6			19.9	10.55 ▴	21.85	19.94	41.514 ▴	21.46	30.3	23.64	21.46	9.754	7	41.25%	30 23
PCB177	30.2			27.7	29.3	27.56	29.36	34.528	31.33	N/A	29.96	29.33	2.623	6	8.75%	N/A
PCB178/129							11.32 •									

TABLE 4 - Ampoule PCBs 4
Results in μg/L

PCB ISOMER	TARGET	9211	#	9212-V	9212-HP	9213A	9213B	9214	9215	9216	MEAN	MEDIAN	SD	n	%RSD	9216 §
PCB180	23.4			21.6	19.2	20.12	16.52	25.109	16.12	27.3	20.85	20.12	4.177	7	20.03%	24.99
PCB187	27.8			21.5	25.4	26.67	20.13	27.554	23.96	29.4	24.94	25.4	3.313	7	13.28%	29.03
PCB189	22.2			N/A	N/A	23.40	23.33	20.429	22.59	27.0	23.35	23.33	2.368	5	10.14%	23.67
PCB190	20.0			16.6	10.55 ▴	18.65	19.66	41.514 ▴	20.58	N/A	21.29	19.26	10.54	6	49.50%	N/A
TOTAL HEPTA	150.2	110	5	87.4	95.0	138.45	140.26	190.648	136.04	114	-					107.9
PCB194	22.0			17.6	18.6	19.77	20.53	21.238	20.39	23.9	20.29	20.39	2.015	7	9.93%	21.95
PCB195	25.4			21.8	21.5	22.41	18.97	62.089	26.75	24.8	28.33	22.41	15.09	7	53.27%	21.62
PCB198	27.8			24.2	23.4	26.78	28.25	37.578	15.65	N/A	25.98	25.49	7.167	6	27.59%	N/A
TOTAL OCTA	75.2	34	3	63.6	63.5	68.96	67.75	120.905	62.79	48.7	-					
PCB209	23.6			17.1	17.8	20.10	18.59	30.874	20.50	22.8	21.11	20.10	4.708	7	22.30%	22.42
TOTAL DECA	23.6	24	1	17.1	17.8	20.10	18.59	30.874	20.50	22.8	-					65.99

#	Laboratory 9211 reported only total congener groups. The values in the adjacent column indicate the total number of analytical peaks detected by this laboratory.
+, &, @, *, $, ▸, ◂, ▴, ▾, «, »	These isomers were reported as co-eluting with another isomer. Results are equally divided between the two isomers.
N/A	Laboratory does not calibrate for this isomer.
ND	Participant did not detect this isomer, which was present in the ampoule, and reported as Not Detected. Participants that did not report a result for an isomer that was present in the ampoule, and did not mark the report form with an "ND", were assigned a "0".
§	Original set of results reported by Laboratory 9216. They had run out of their own calibration standards when the ampoules were received. To try and meet the study deadline, they borrowed calibration standards that had been cross-checked to the same external sources that they used. When they received the table of results from this study, they did felt that their performance could have been better. Upon comparing the borrowed standards with their own new standards, Laboratory 9216 felt that the borrowed standards were the source of variability in their performance. They reanalysed the ampouled solutions using their new calibration standards. The revised results are used in the calculations of mean and standard deviation.
* *	Distribution of results bimodal. Mean, Median, SD and %RSD unreliable.
INT	Interference(s) for this isomer. Could not quantitate the peak area.

Laboratories 9212 and 9213 provided two sets of results using two different instruments. See Table 6 for description.

TABLE 5: Distribution of PCB Isomers on Participants' Target Lists

Number on Target List	PCB Isomer
All participants (6)	PCB18 PCB31 PCB40 PCB44 PCB49 PCB52 PCB77 PCB101 PCB105 PCB114 PCB118 PCB128 PCB137 PCB138 PCB153 PCB156 PCB170 PCB180 PCB187 PCB194 PCB195 PCB209
Target List of 1 Participant Only	PCB15 PCB61 PCB75 PCB166 PCB169
Not on any Participant's Target List	PCB123 PCB126 PCB155

TABLE 6: Instruments and GC Columns

Lab Code	9211	9212-V	9212-HP	9213	9214	9215	9216
Instrument	GC/MSD: HP	GC/ECD: Varian 3400	GC/ECD: HP5890	A: GC/ECD: Varian 3400 B: GC/ECD: Varian Vista 6000	GC/ECD: HP5890	GC/ECD: HP5890	GC/ECD: HP3890
Column	DB-5, 30 m	DB-5, 60 m DB-1701, 60 m	DB-5, 60 m DB-1701, 60 m	DB-5, 60 m	DB-5, 30 m	DB-5, 60 m DB-17, 30 m	DB-1, 30 m DB-5, 30 m

NOTE 1: Laboratory 9213 analyzed the ampoules using the Varian 3400 with a SPI (Septum Programmable Injector). They repeated their analysis of the PCB ampoules using the Varian Vista 6000. Both sets of results are included in Tables 1-4 as marked.

NOTE 2: Laboratory 9216 had run out of their own calibration standards when the ampoules were received. To try and meet the study deadline, they borrowed calibration standards that had been cross-checked to the same external sources that they used. When they received the original table of results, they did not feel that their performance could have been better. Upon investigating the borrowed standards with their new standards, they felt that the borrowed standards were the source of variability in their performance. They reanalysed the ampouled solutions using their new calibration standards. Both sets of results are included in Tables 1-4 as marked.

TABLE 7: Distribution of Participants' Results Relative to Target

Lab ID Code	9212-V		9212-HP		9213A		9213B		9214		9215		9216	
Percent of Target	n	%	n	%	n	%	n	%	n	%	n	%	n	%
<60%	10	11.6%	10	11.6%	14	12.5%	20	17.2%	17	12.7%	8	6.1%	5	10%
60-75%	7	8.1%	2	2.3%	4	3.6%	19	16.4%	11	8.2%	12	9.2%	0	0%
75-90%	47	54.7%	29	33.7%	20	17.9%	35	30.2%	20	14.9%	34	26.0%	0	0%
90-110%	17	19.8%	40	46.5%	50	44.6%	24	20.7%	36	26.9%	52	39.7%	24	48%
110-130%	4	4.7%	3	3.5%	13	11.6%	8	6.9%	12	9.0%	22	16.8%	15	30%
>130%	1	1.2%	2	2.3%	11	9.8%	10	8.6%	38	28.4%	3	2.3%	6	12%

n - number of results falling in range (includes results from all four ampoules)

TABLE 8 - Performance of Laboratory 9211

CONGENER GROUP	TARGET	RESULT	% OF TARGET	# PEAKS PRESENT	# PEAKS REPORTED
IADN PCBs 1					
TOTAL MONO	232	210	90.5%	1	1
TOTAL DI	311.3	260	83.5%	2	2
TOTAL TRI	46.6	41	88.0%	4	5
TOTAL TETRA	75.5	78	103.3%	7	7
TOTAL PENTA	44.4	34	76.6%	4	4
TOTAL HEXA	79.7	81	101.6%	5	5
TOTAL HEPTA	13.3	6.9	51.9%	1	1
TOTAL OCTA	11	6.6	60.0%	1	1
IADN PCBs 2					
TOTAL DI	474	320	67.5%	2	2
TOTAL TRI	55.9	43	76.9%	5	5
TOTAL TETRA	52.4	60	114.5%	5	4
TOTAL PENTA	48.1	38	79.0%	4	4
TOTAL HEXA	47	42	89.4%	4	4
TOTAL HEPTA	26.2	20	76.3%	2	2
TOTAL OCTA	25.7	6	23.3%	1	1
TOTAL DECA	11.8	13	110.2%	1	1
IADN PCBs 3					
TOTAL DI	432	360	83.3%	2	2
TOTAL TRI	58.4	50	85.6%	5	5
TOTAL TETRA	63.5	79	124.4%	5	6
TOTAL PENTA	48	40	83.3%	4	4
TOTAL HEXA	69.2	72	104.0%	5	5
TOTAL HEPTA	35.6	30	84.3%	3	3
TOTAL OCTA	12.7	6.2	48.8%	1	1
IADN PCBs 4					
TOTAL MONO	348	330	94.8%	1	1
TOTAL DI	1809	1490	82.4%	5	4
TOTAL TRI	344.4	270	78.4%	15	12
TOTAL TETRA	382.8	440	114.9%	17	13
TOTAL PENTA	281	220	78.3%	12	12
TOTAL HEXA	391.8	380	97.0%	14	11
TOTAL HEPTA	150.2	110	73.2%	6	5
TOTAL OCTA	75.2	34	45.2%	3	3
TOTAL DECA	23.6	24	101.7%	1	1

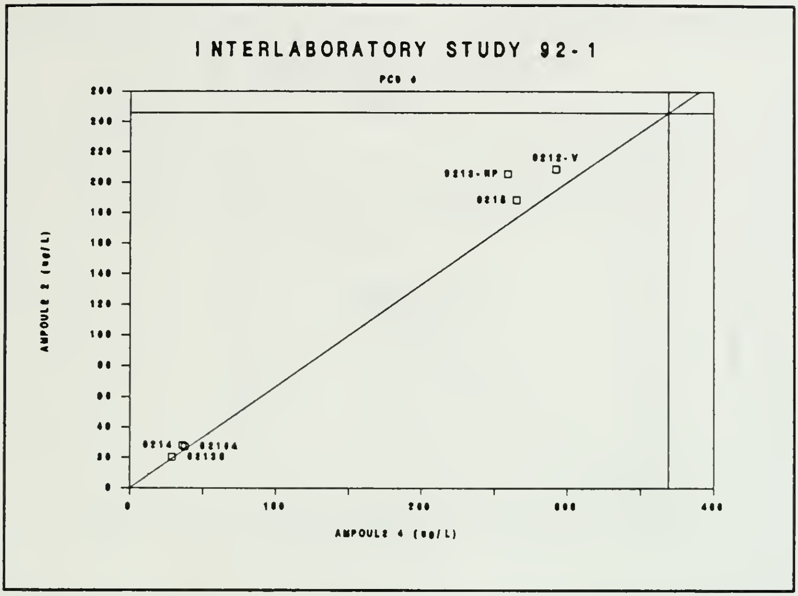

Figure 1 - PCB4

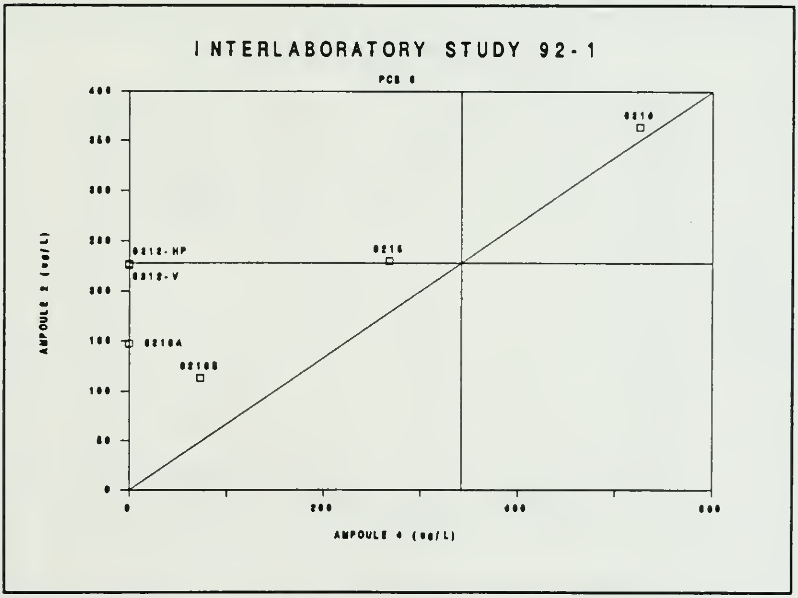

Figure 2 - PCB8

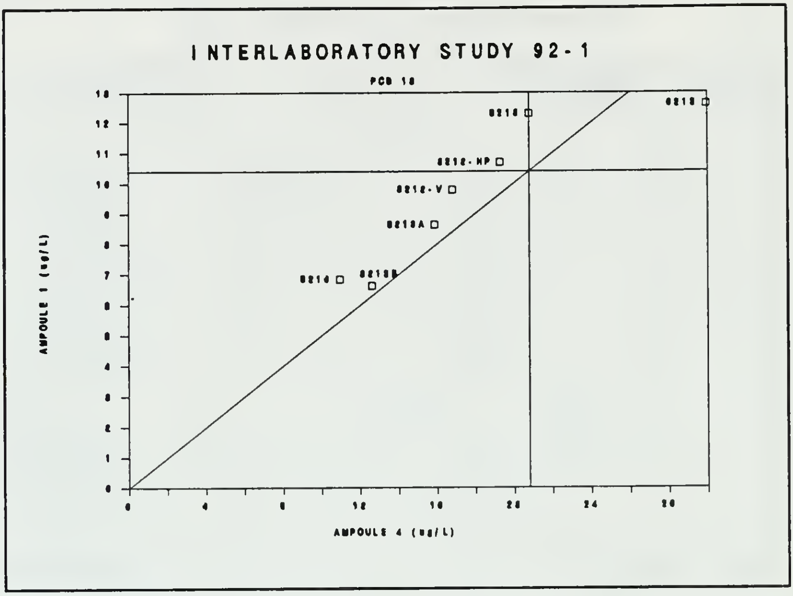

Figure 3 - PCB18

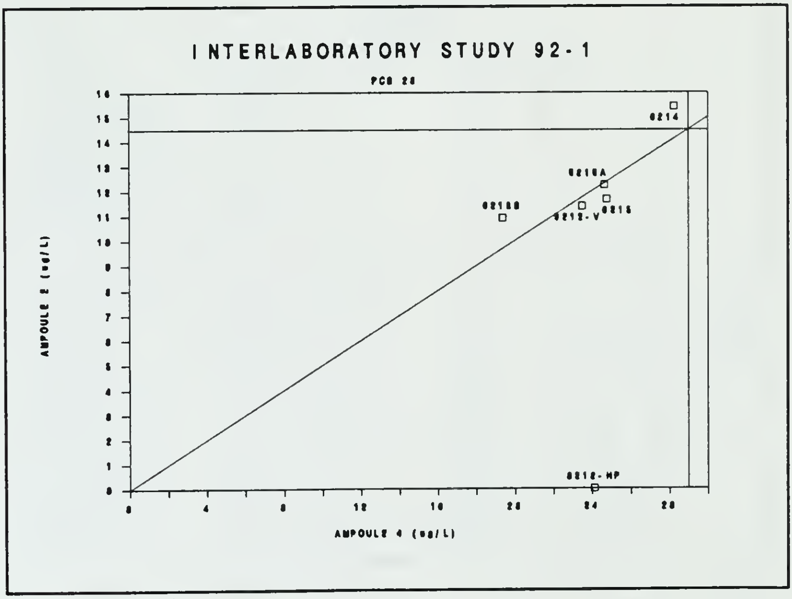

Figure 4 - PCB28

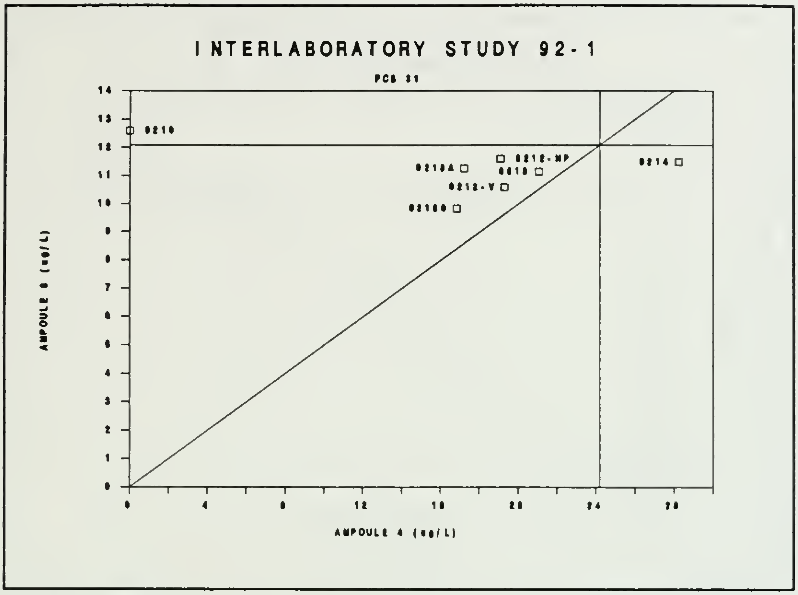

Figure 5 - PCB31

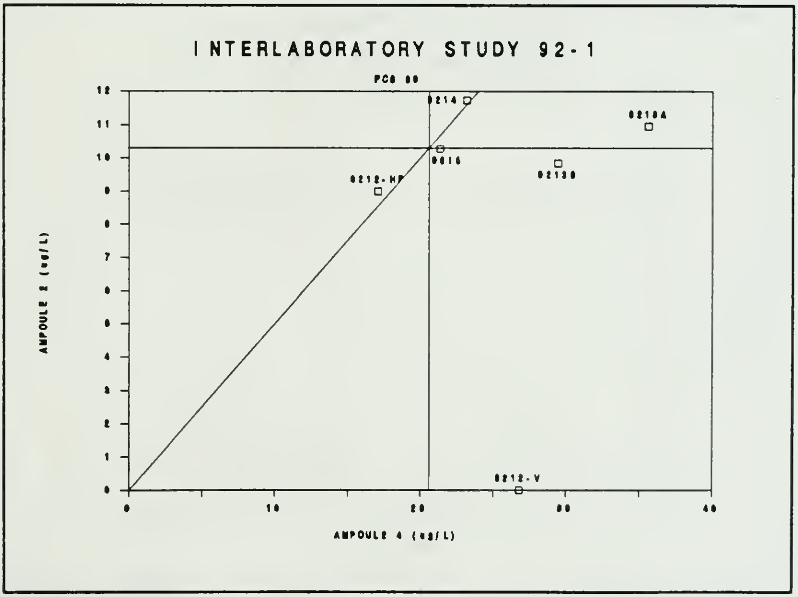

Figure 6 - PCB33

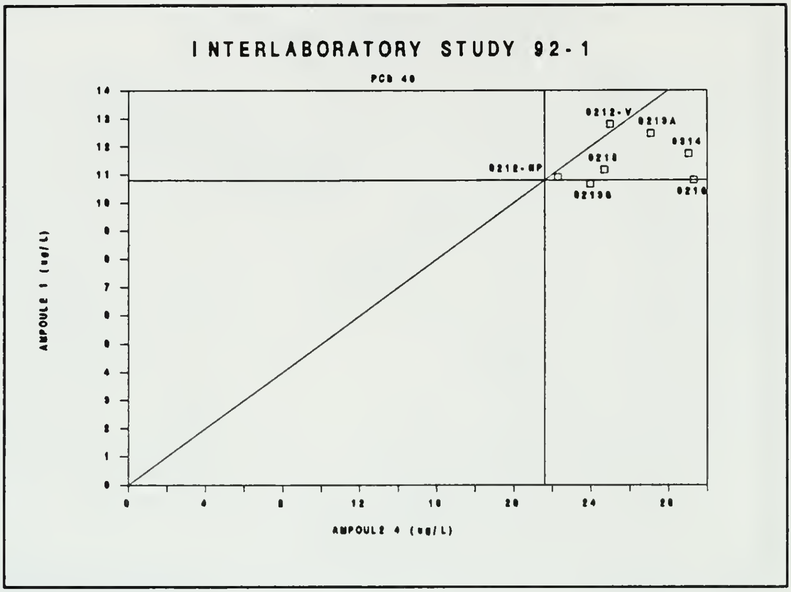

Figure 7 - PCB40

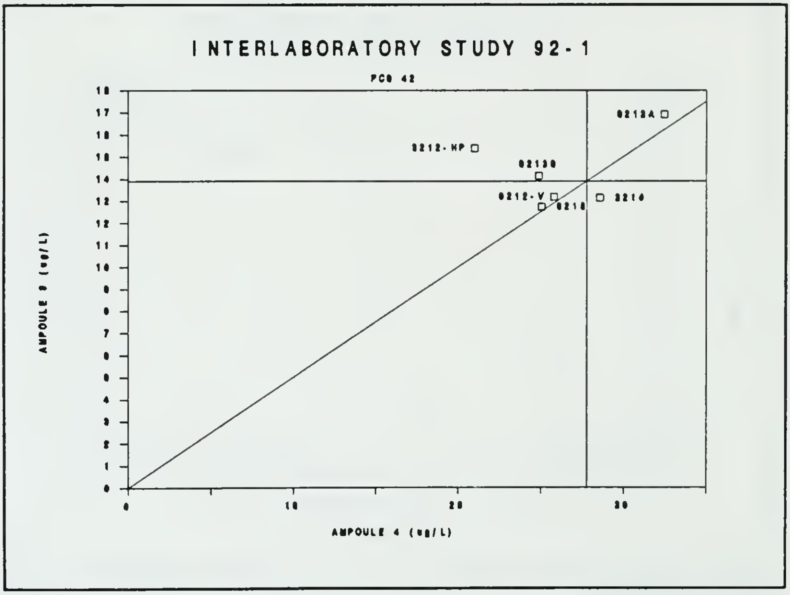

Figure 8 - PCB42

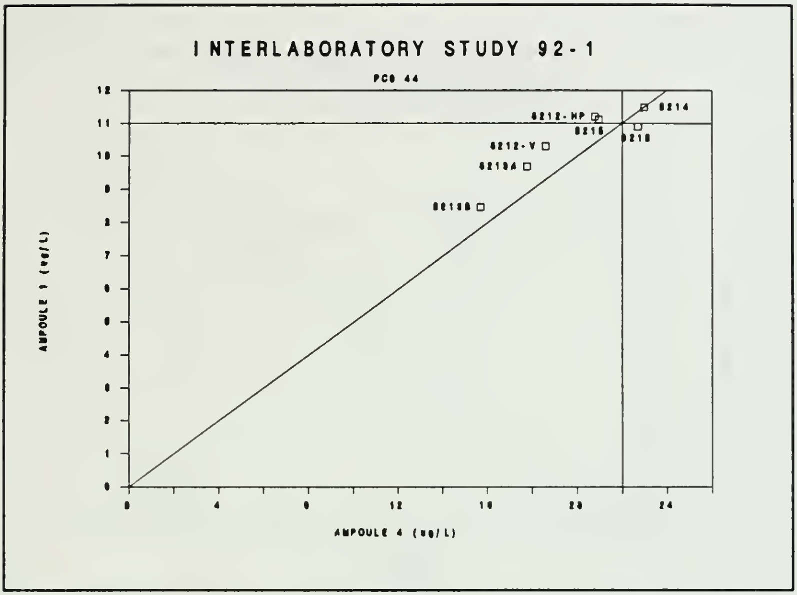

Figure 9 - PCB44

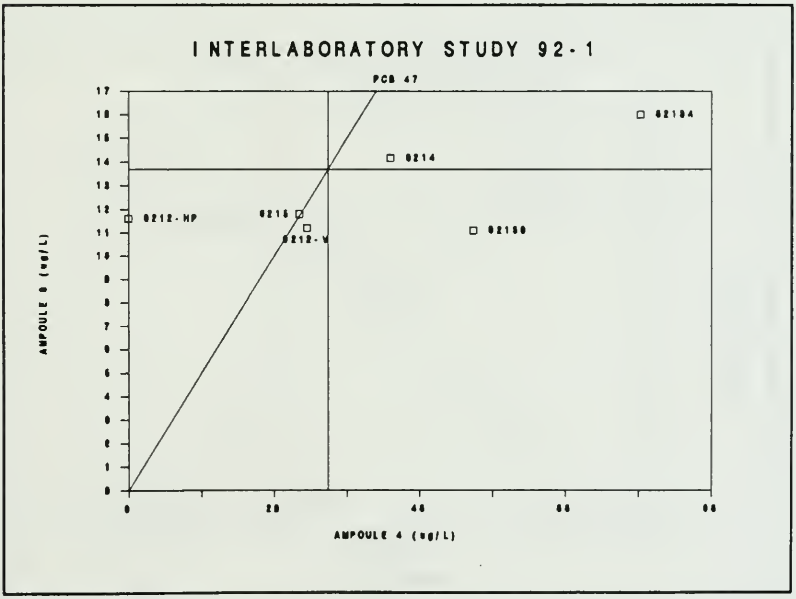

Figure 10 - PCB47

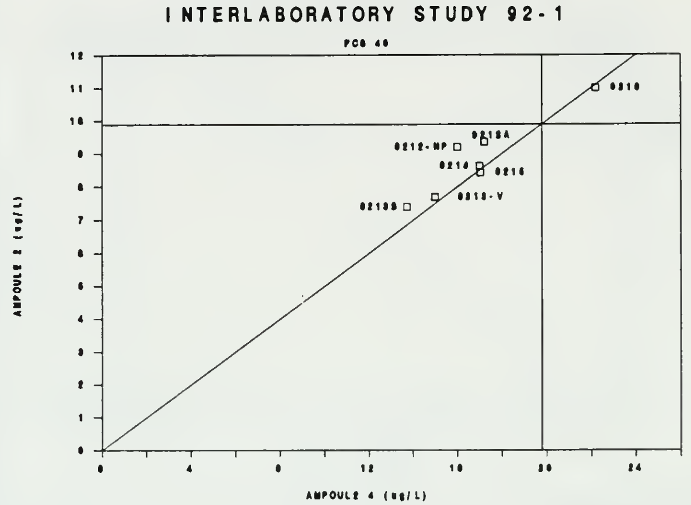

Figure 11 - PCB49

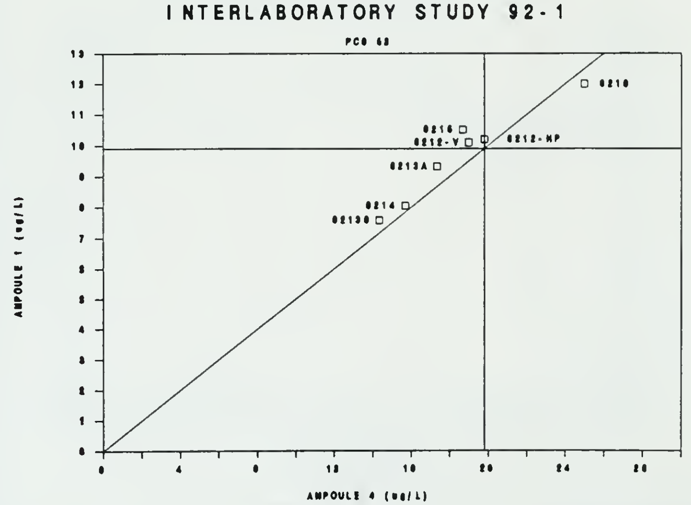

Figure 12 - PCB52

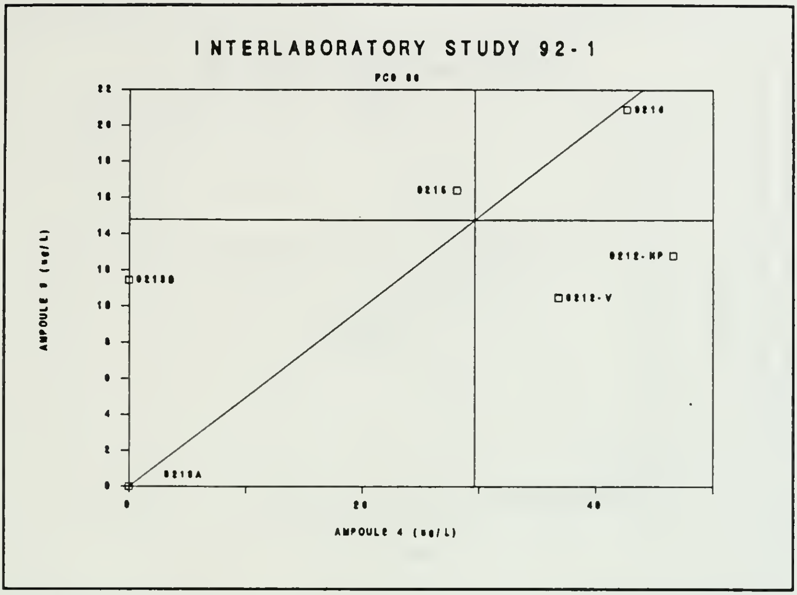

Figure 13 - PCB66

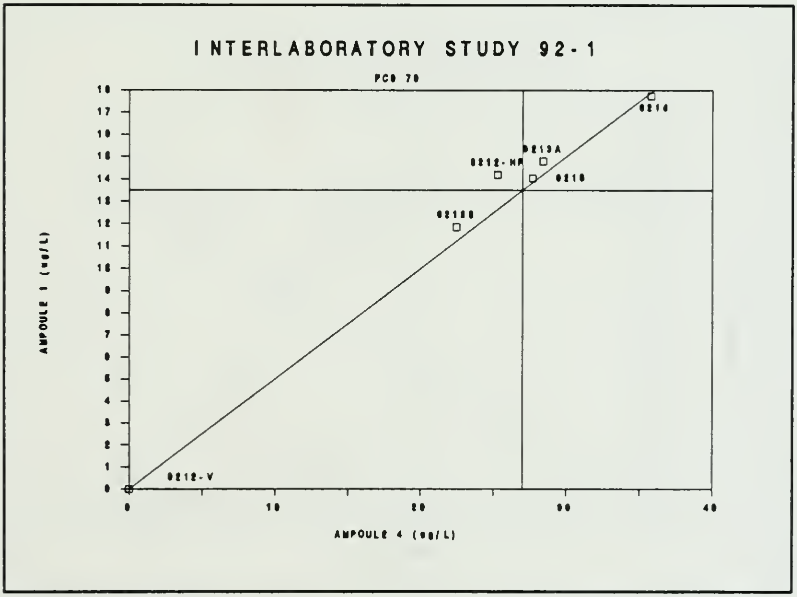

Figure 14 - PCB70

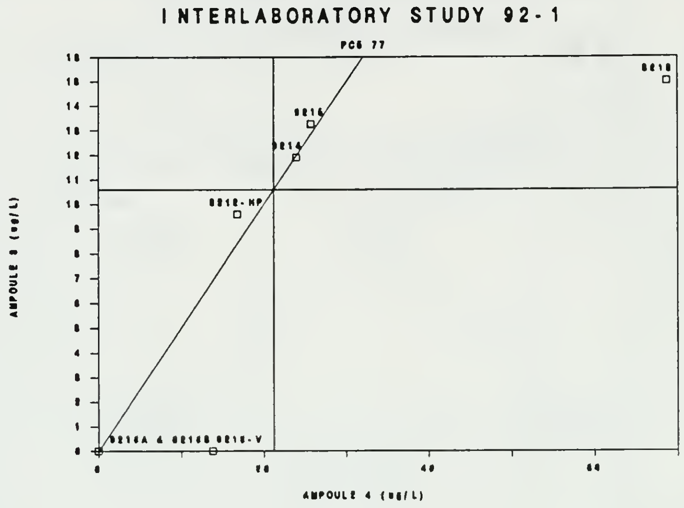

Figure 15 - PCB77

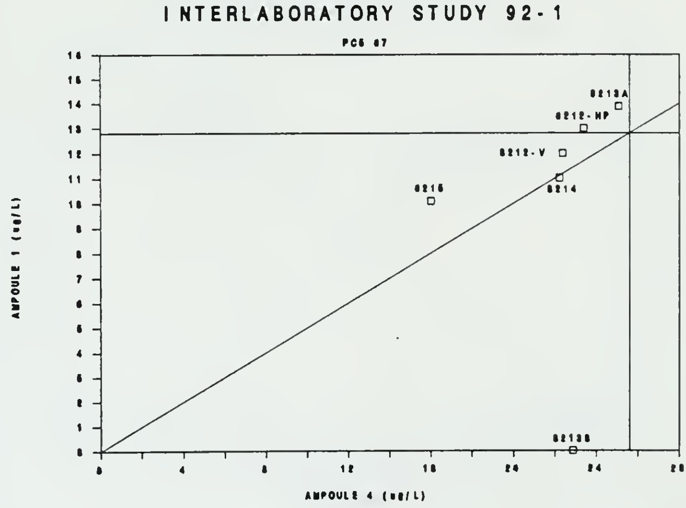

Figure 16 - PCB97

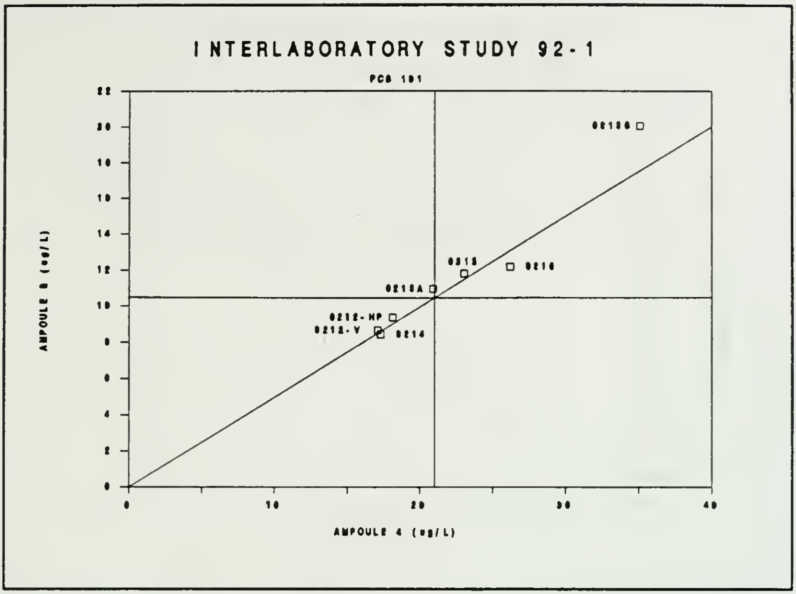

Figure 17 - PCB101

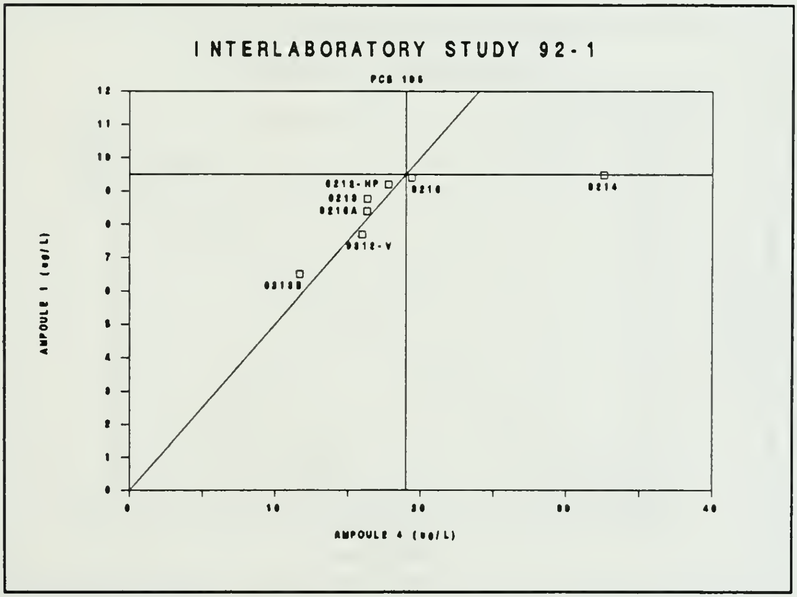

Figure 18 - PCB105

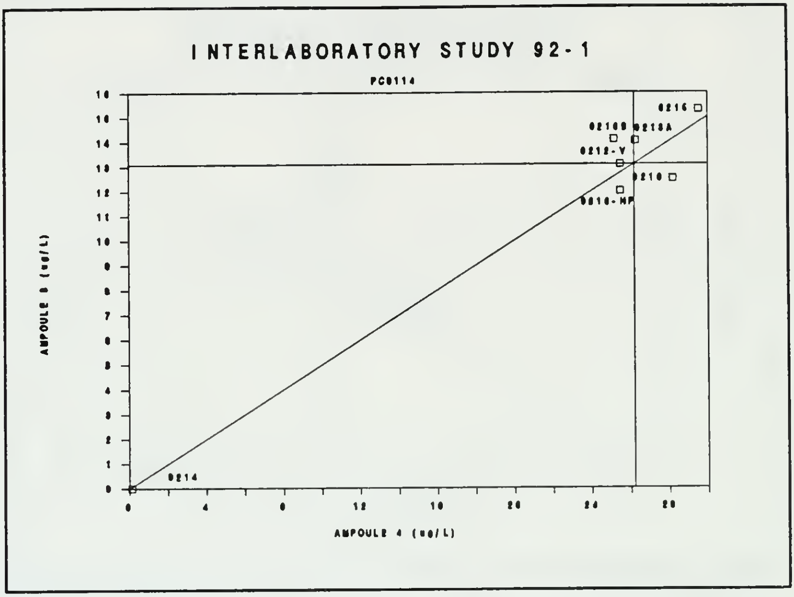

Figure 19 - PCB114

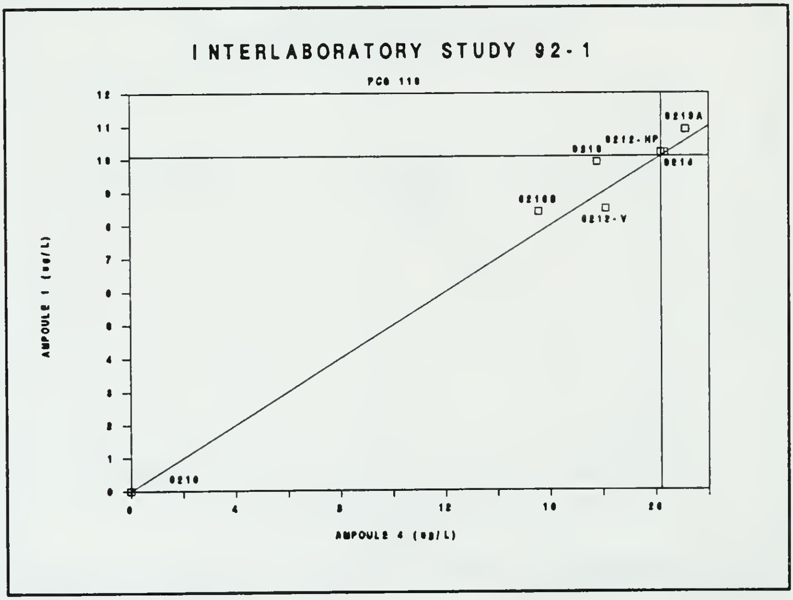

Figure 20 - PCB118

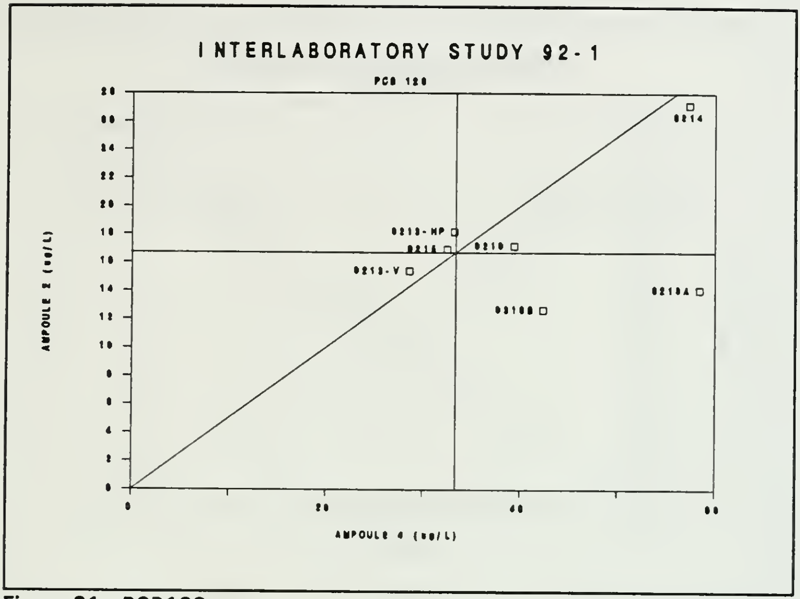

Figure 21 - PCB128

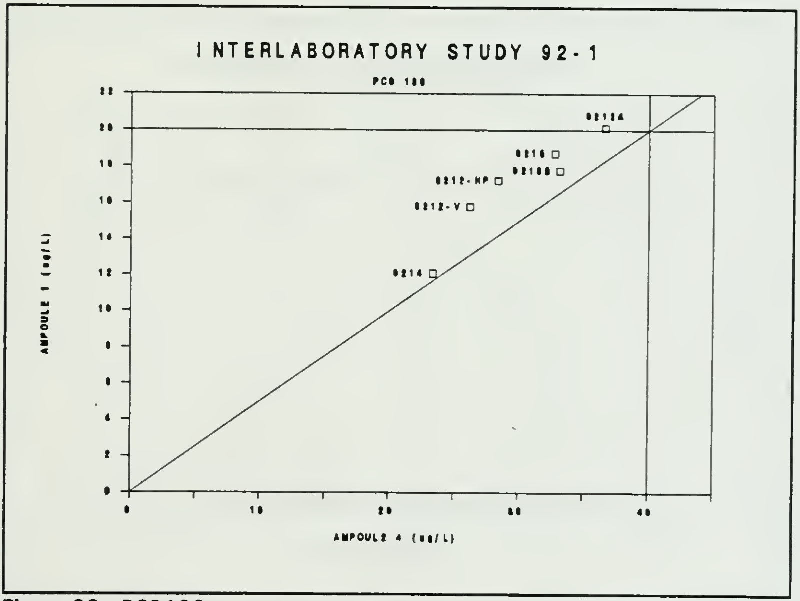

Figure 22 - PCB136

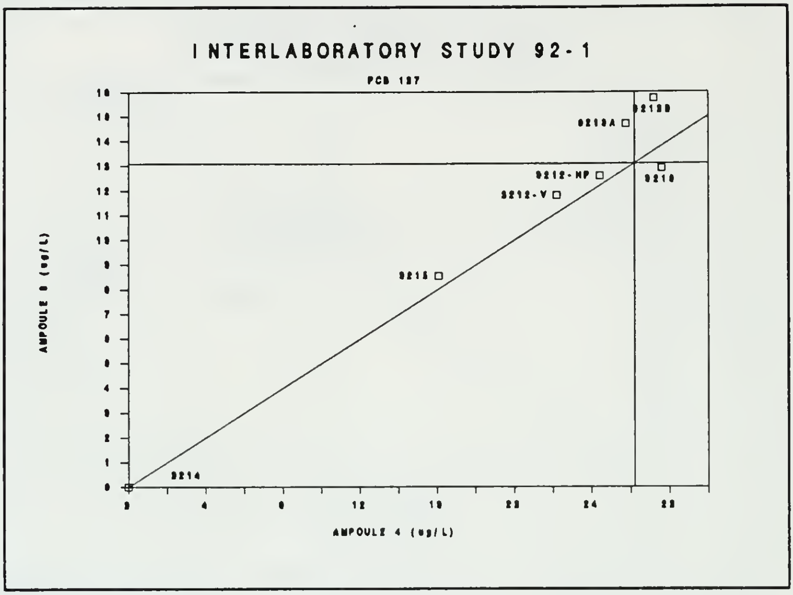

Figure 23 - PCB137

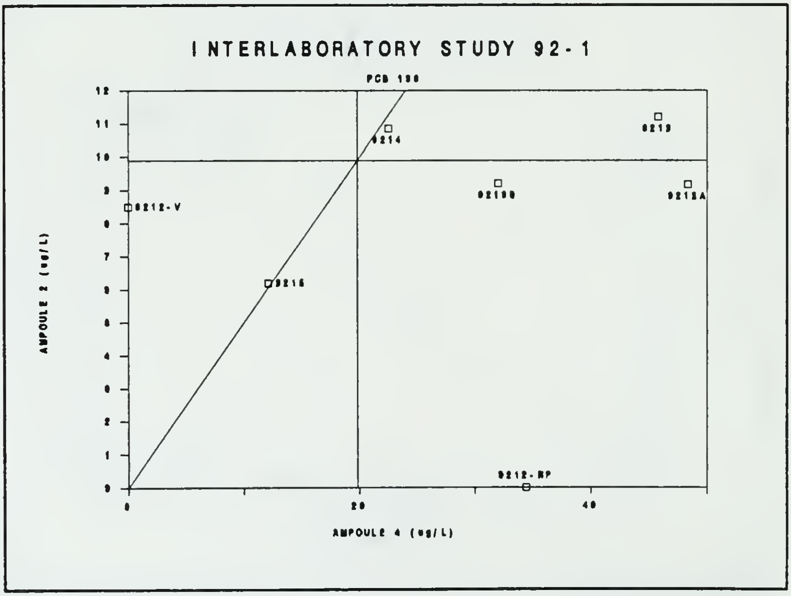

Figure 24 - PCB138

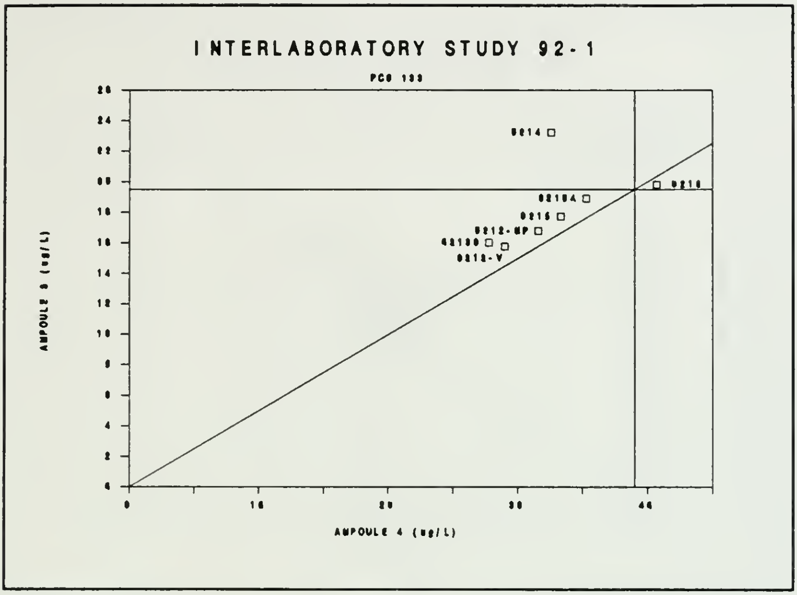

Figure 25 - PCB153

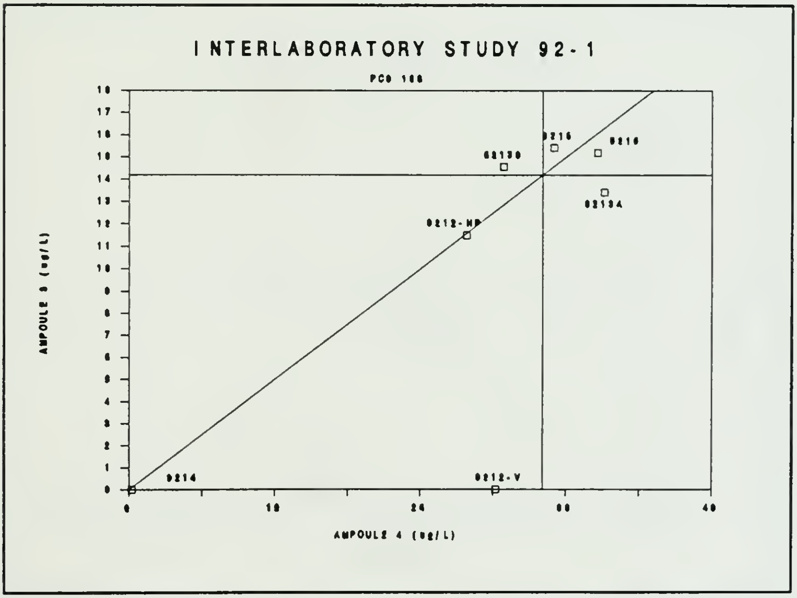

Figure 26 - PCB156

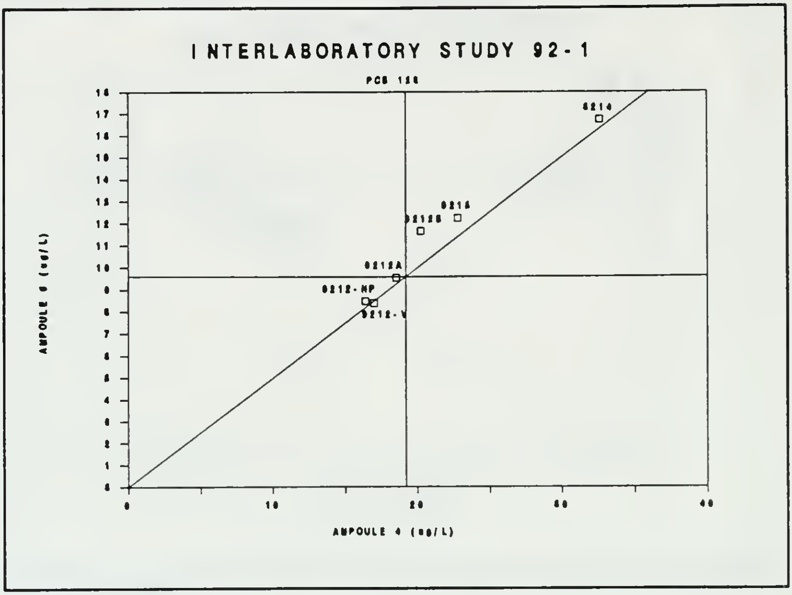

Figure 27 - PCB158

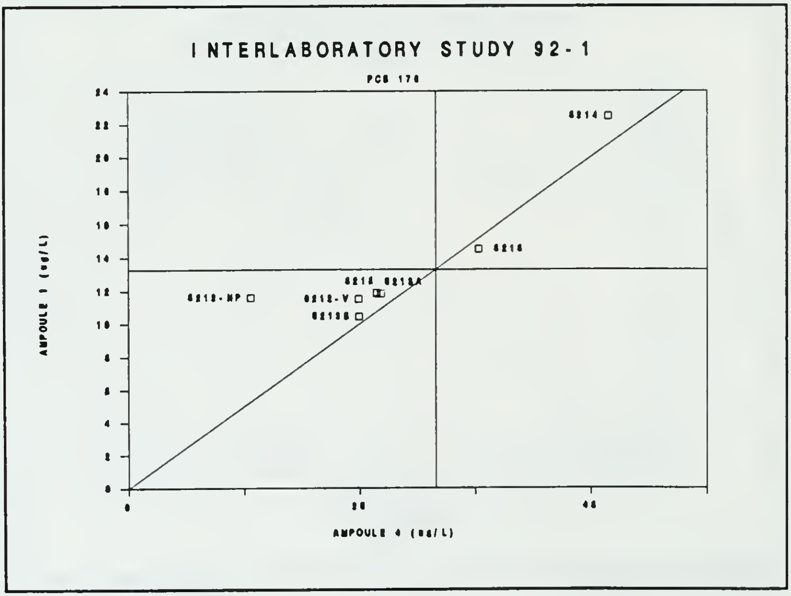

Figure 28 - PCB170

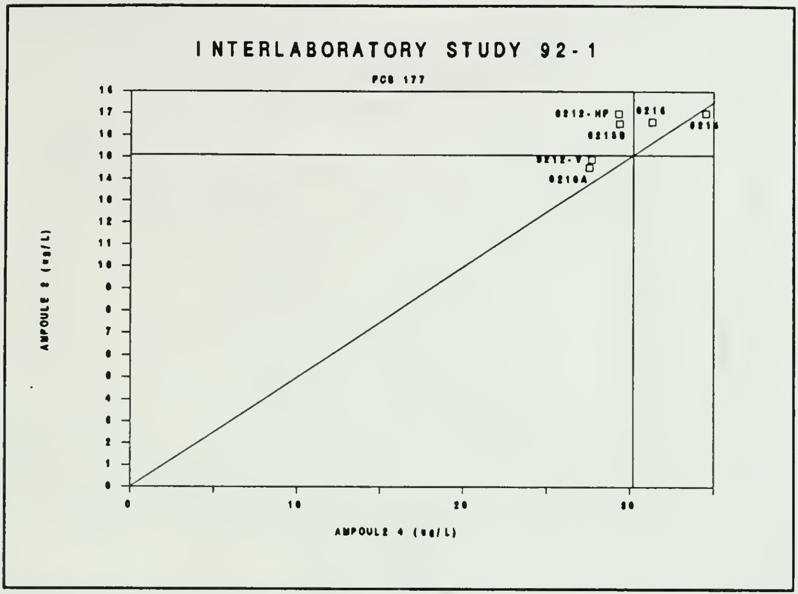

Figure 29 - PCB177

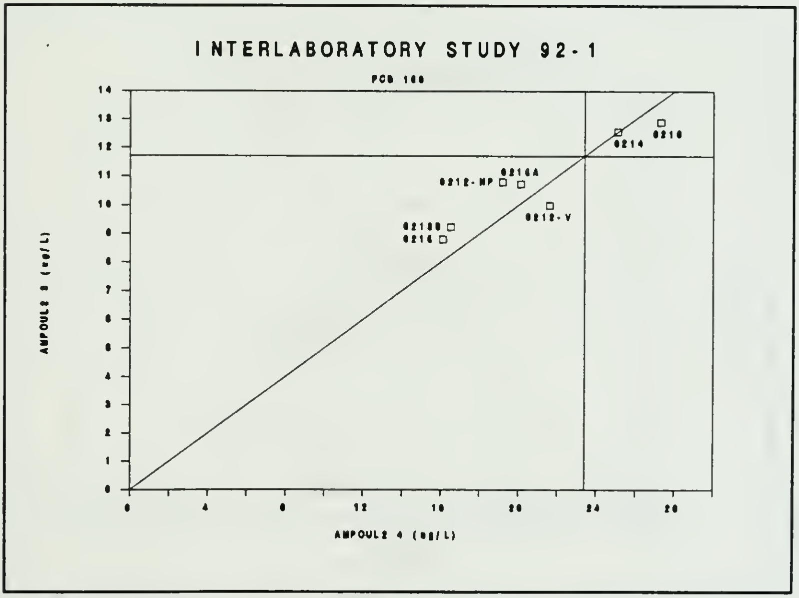

Figure 30 - PCB180

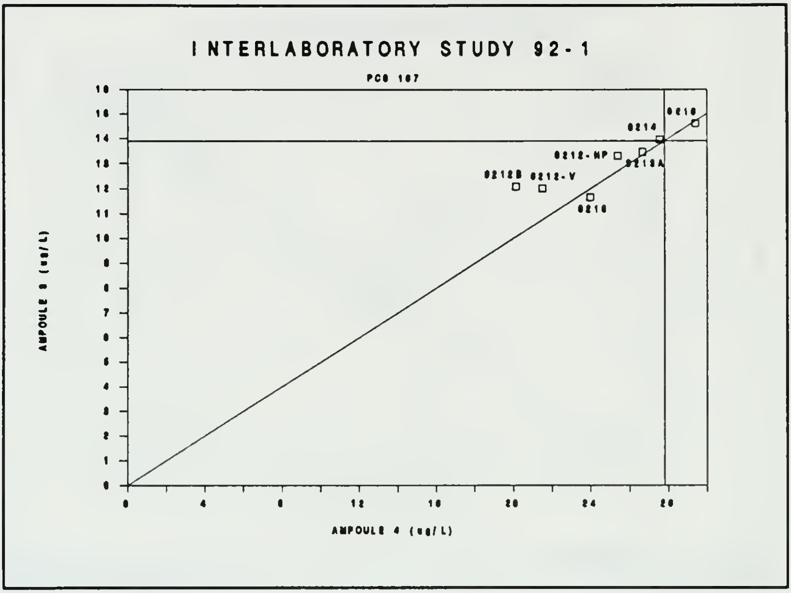

Figure 31 - PCB187

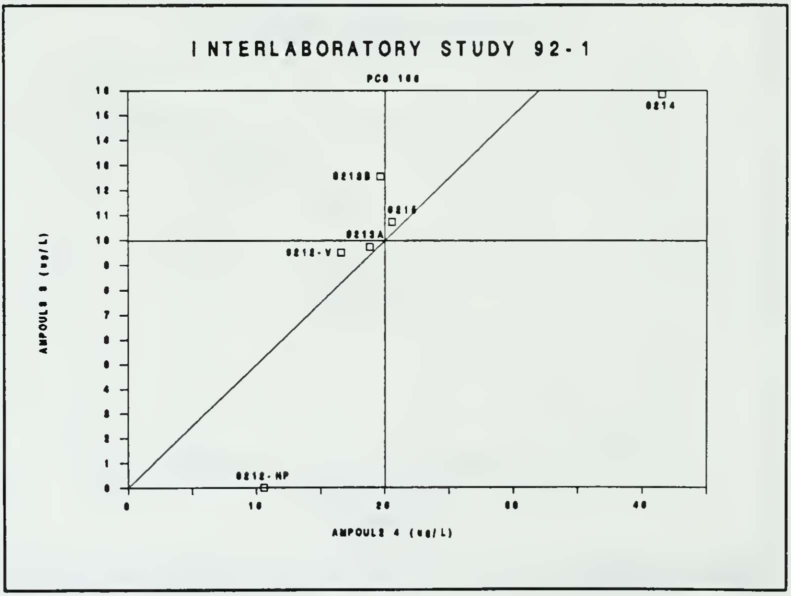

Figure 32 - PCB190

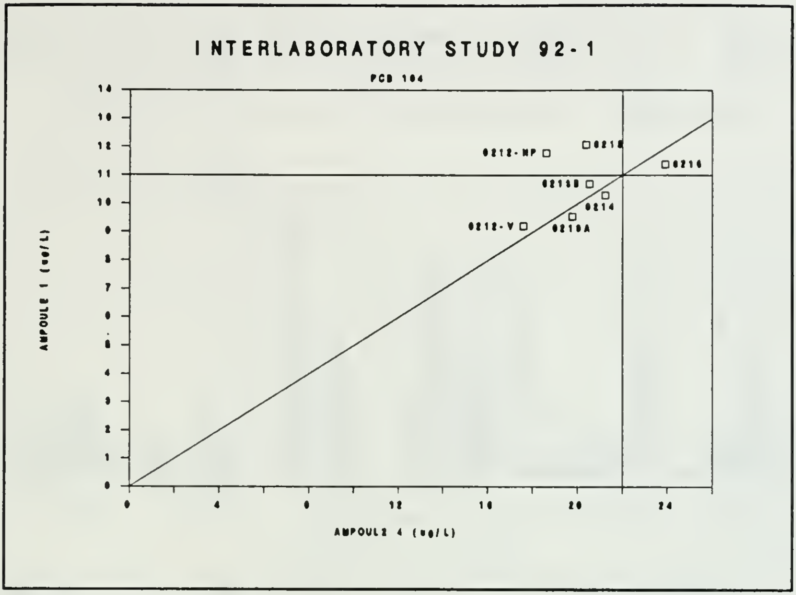

Figure 33 - PCB194

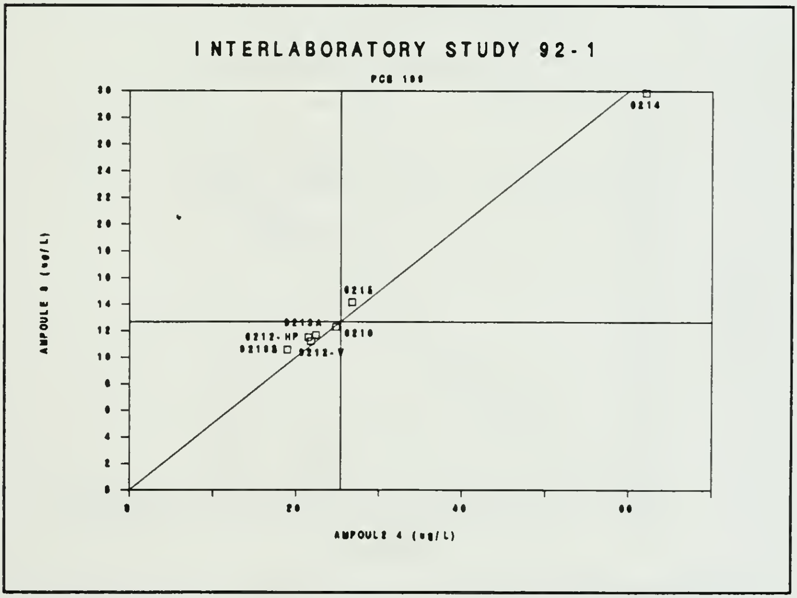

Figure 34 - PCB195

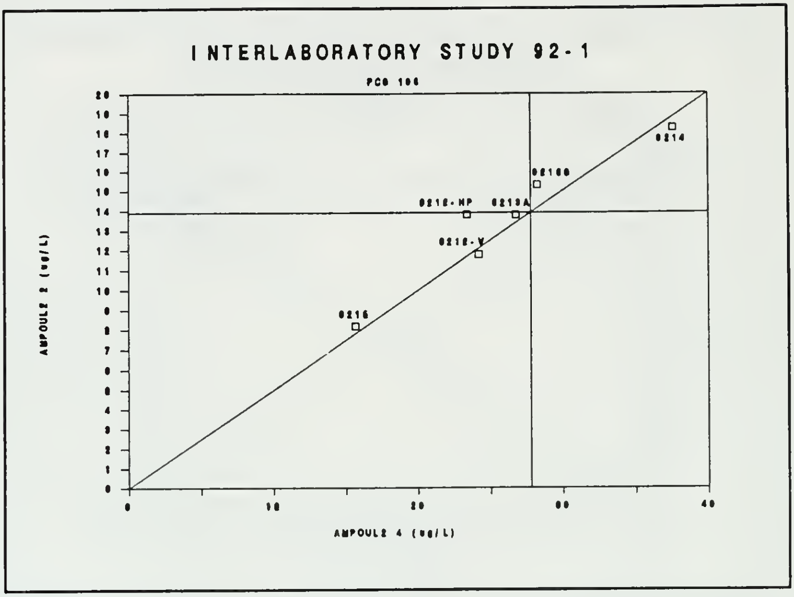

Figure 35 - PCB198

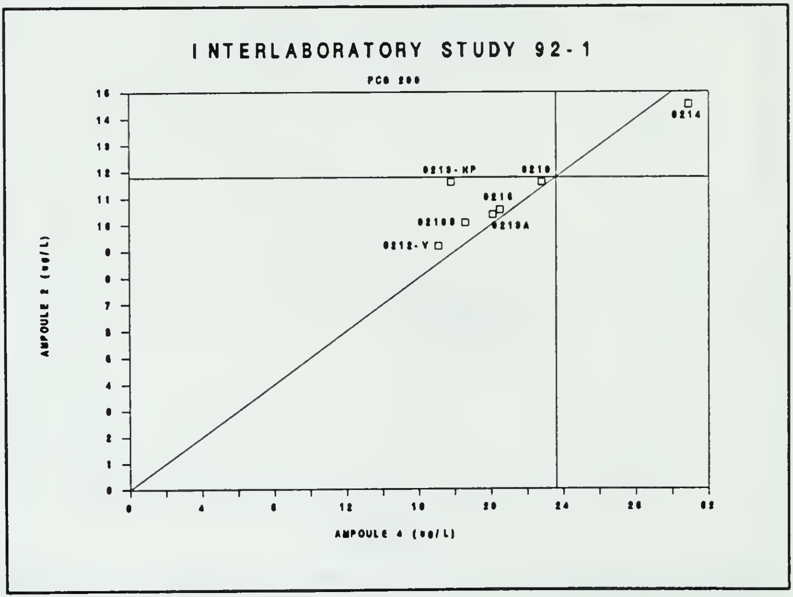

Figure 36 - PCB209

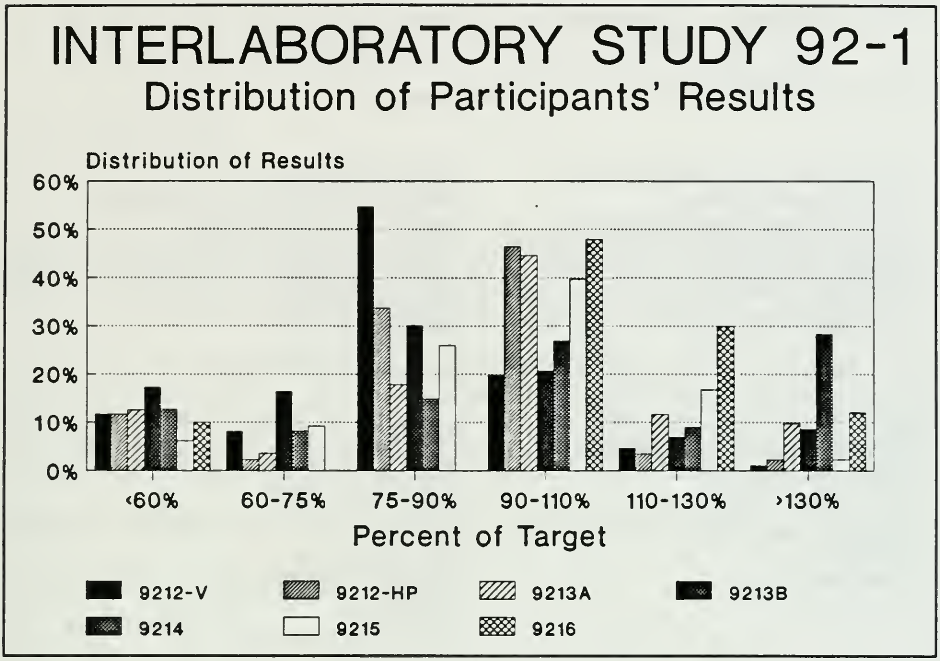

Figure 37 - Distribution of Participants' Results (PCB Isomers)

8 APPENDIX 2 - PARTICIPANTS AND CORRESPONDENCE

List of Participants

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Environmental Technology Centre
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Gloucester, Ontario
K1G 3N3
(613) 990-8560

Quality Management Office

September 28, 1992

Dear Interlaboratory Study 92-1 Participant,

Please find enclosed four 5 mL ampoules for the analysis of Polychlorinated Biphenyl Isomers (PCB's). The ampoules are labelled IADN PCBs 1, IADN PCBs 2, IADN PCBs 3, and IADN PCBs 4. The solvent is Iso-octane. If you are missing any of the ampoules or they have broken in transit, please contact me at (416) 235-5842 immediately for replacement.

The ampoules are ready for direct instrumental analysis. Break open the ampoule on the scored mark and transfer the contents to the appropriate sample container for your analytical system. No dilutions should be required, but if you do so, please mark the dilution factor used on the accompanying report form. The parameters present are indicated on the form. Please note that each ampoule does not necessarily contain all of the isomers.

Please report all results on the accompanying form by October 16, 1992.

Thank you for your participation in this study.

Your identification code is:

Sylvia Cussion
Laboratory Quality Audit Scientist
(416) 235-5842
FAX (416) 235-6110

INTERLABORATORY STUDY 92-1

PCB ISOMERS FOR THE INTEGRATED ATMOSPHERIC DEPOSITION NETWORK

Identification Code:

Units:

PCB ISOMER	IADN PCBs 1	IADN PCBs 2	IADN PCBs 3	IADN PCBs 4
PCB3				
PCB4				
PCB5				
PCB7				
PCB8				
PCB15				
PCB16				
PCB17				
PCB18				
PCB19				
PCB22				
PCB24				
PCB25				
PCB26				
PCB27				
PCB28				
PCB29				
PCB31				
PCB32				
PCB33				
PCB37				
PCB40				
PCB41				
PCB42				
PCB44				
PCB48				
PCB49				

PCB ISOMER	IADN PCBs 1	IADN PCBs 2	IADN PCBs 3	IADN PCBs 4
PCB52				
PCB53				
PCB61				
PCB66				
PCB70				
PCB71				
PCB74				
PCB75				
PCB77				
PCB81				
PCB84				
PCB95				
PCB97				
PCB100				
PCB101				
PCB105				
PCB110				
PCB114				
PCB118				
PCB119				
PCB123				
PCB126				
PCB128				
PCB136				
PCB137				
PCB138				
PCB149				
PCB153				
PCB155				
PCB156				
PCB157				
PCB158				
PCB163				

PCB ISOMER	IADN PCBs 1	IADN PCBs 2	IADN PCBs 3	IADN PCBs 4
PCB166				
PCB167				
PCB169				
PCB170				
PCB177				
PCB180				
PCB187				
PCB189				
PCB190				
PCB194				
PCB195				
PCB198				
PCB209				

INSTRUMENT AND DETECTOR USED FOR ANALYSIS:

125 Resources Rd.
Etobicoke, Ontario, M9P 3V6
Phone: (416) 235-5842
FAX: (416) 235-6107

February 25, 1993

TO: PARTICIPANTS OF INTERLABORATORY STUDY 92-1

Thank you for your participation in Interlaboratory Study 92-1 for the analysis of PCB Isomers in ampouled standards. This study was in support of the Integrated Atmospheric Deposition Network (IADN) program. I apologize for the delay in reporting results, but one participant did not report their final results until last week.

The results are provided in the attached tables. Target values are provided. Please inform me of any transcription errors by March 12, 1993.

Each participant received a set of ampoules prepared from the same stock solution, prepared in iso-octane.

One participant did not report individual isomer results, but only total congener groups. To provide a comparison with the other participants, the results for each congener group have been summed for all participants. The target value for each congener group sum has also been provided.

The Quality Assurance Working Group of the Canada-Ontario Agreement (COA) has been the directing force for these interlaboratory studies. They wish to know the identities of the participating laboratories for the purposes of data comparison for IADN. The Program Managers and Principle Investigators (U.S. and Canadian) for IADN also wish to have the laboratories identified. As the original invitation for this study indicated that laboratory codes were confidential, I am asking each participant to give me permission to reveal their identities to these groups of data users. Please notify me in writing with your permission to reveal your study code.

A date has not yet been set for the next set of ampouled standards in this series of IADN studies, but hopefully will take place in April or early May. A letter will be sent out giving several weeks notice.

Your identification code is:

Sylvia Cussion
Laboratory Quality Audit Scientist